VOL. LVI, NO. 4, APRIL, 1961

THE Journal OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

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Vol. LVI APRIL 1961 No. 4

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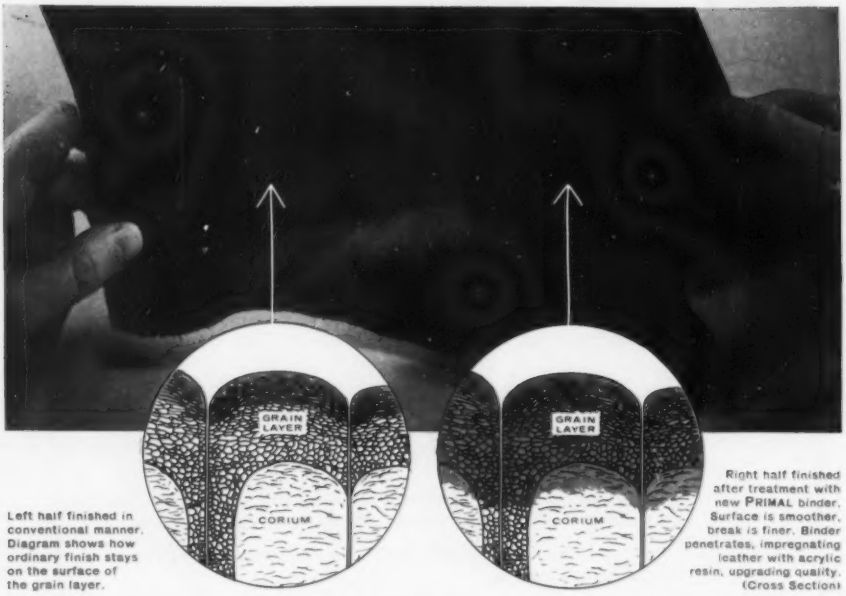

Left half finished in conventional manner. Diagram shows how ordinary finish stays on the surface of the grain layer.

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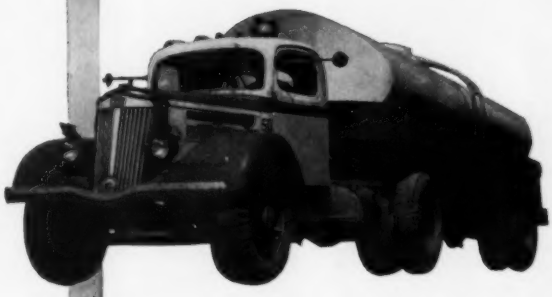

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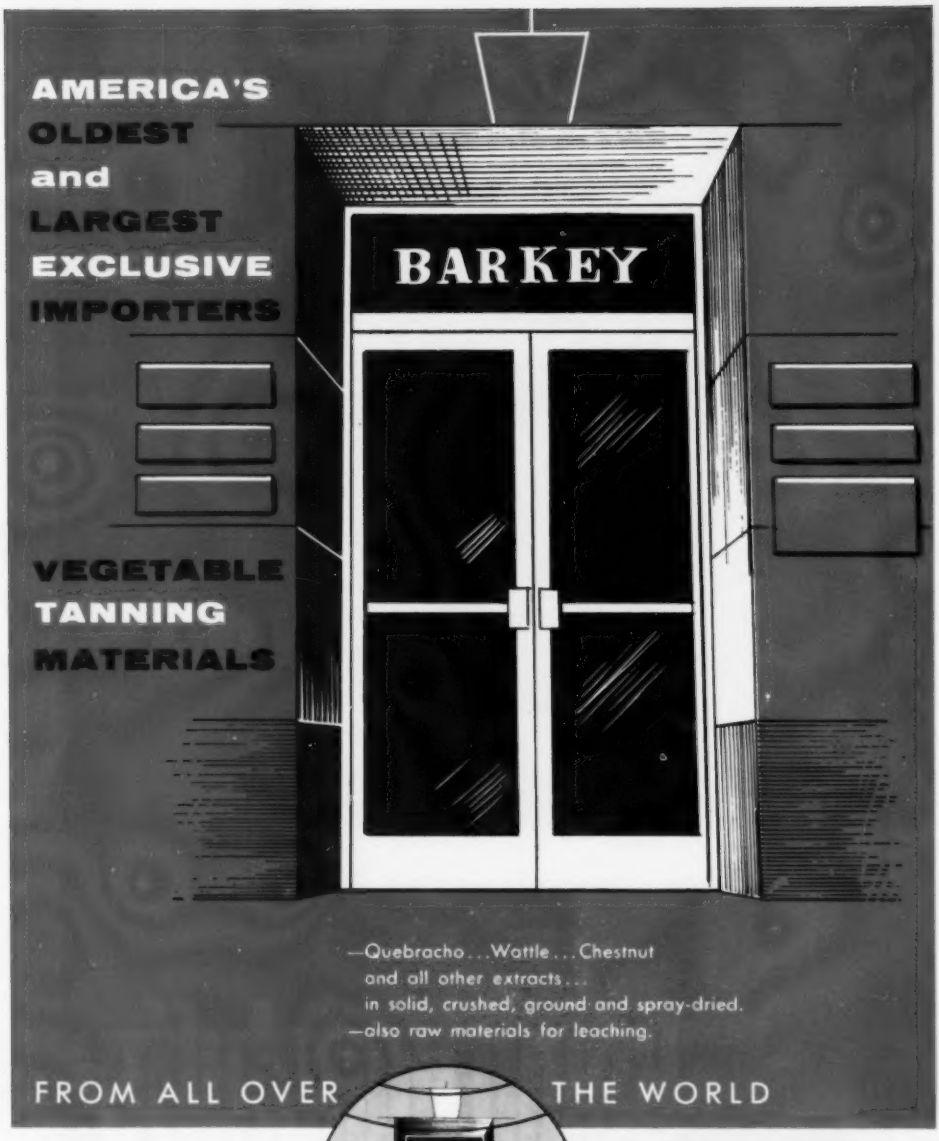

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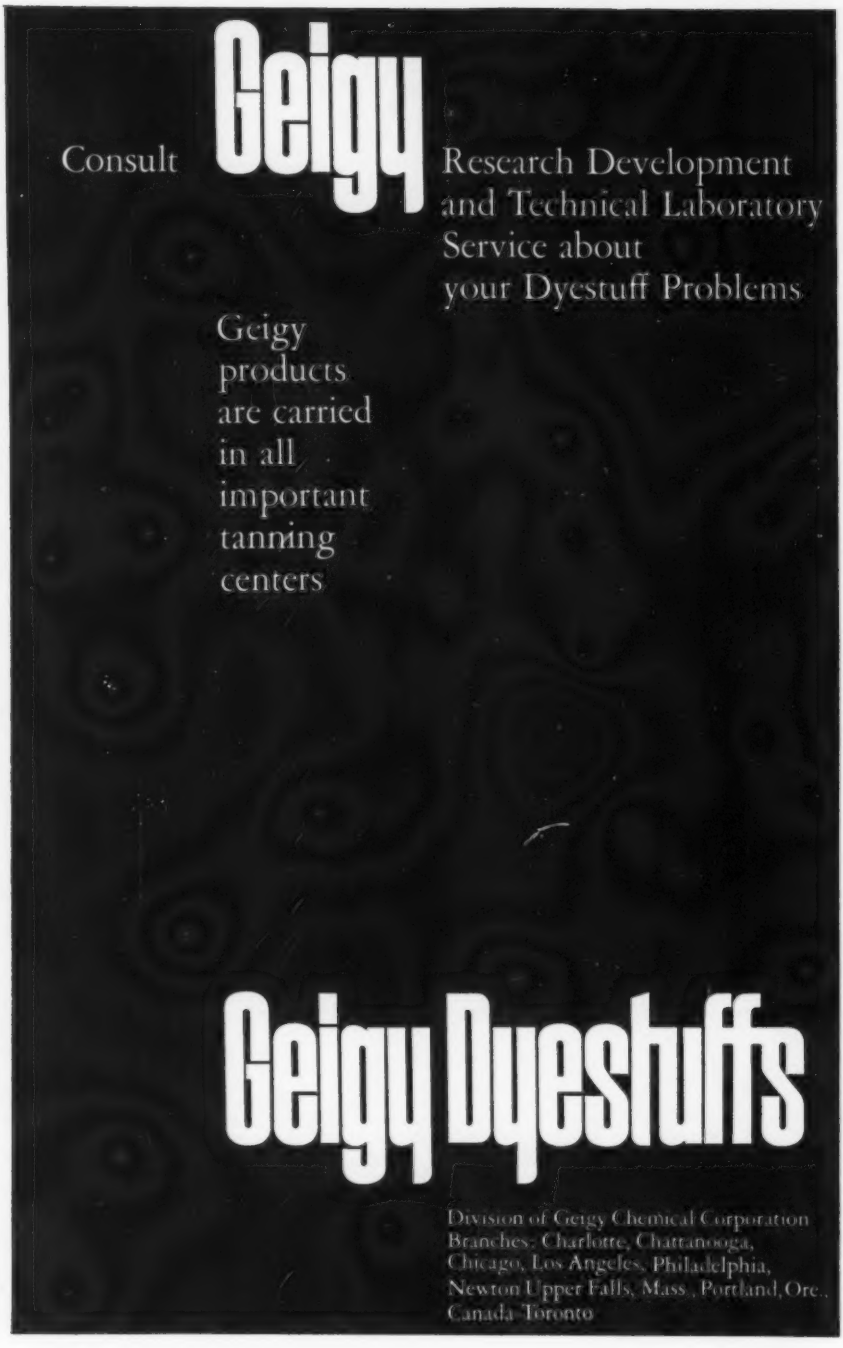
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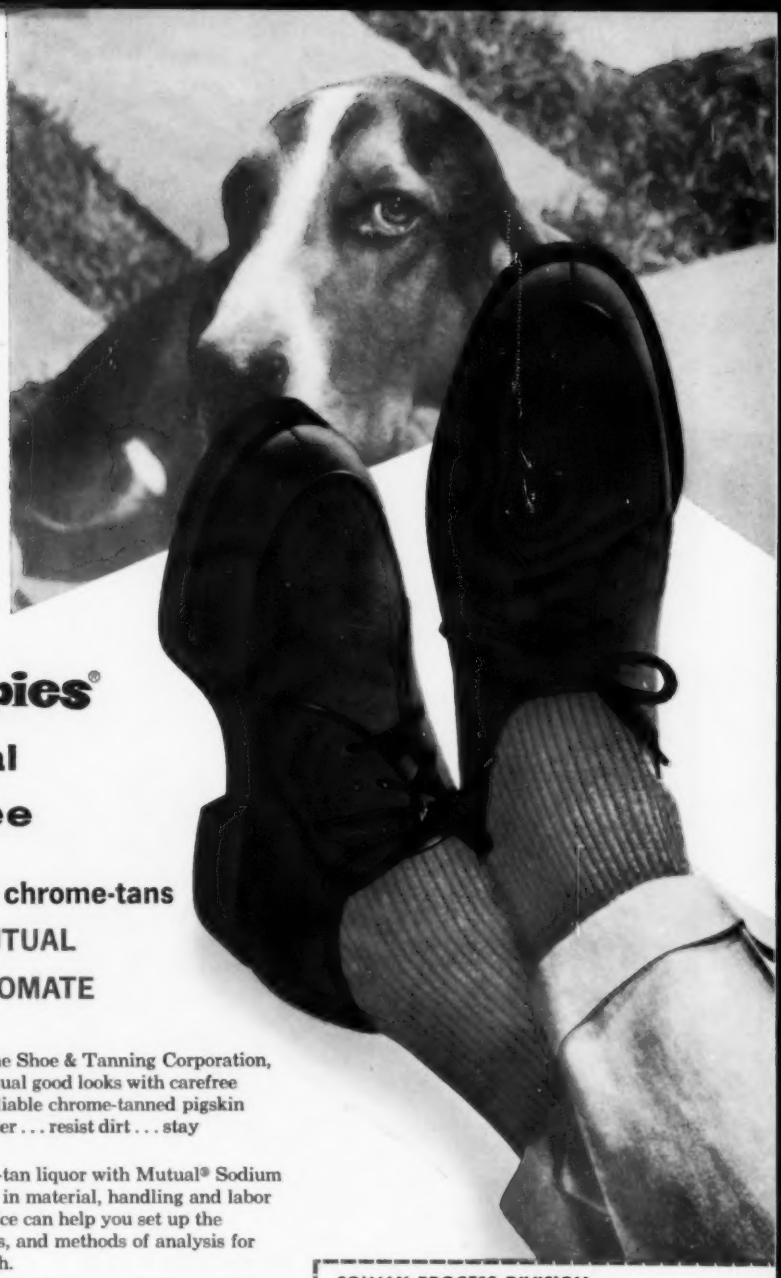

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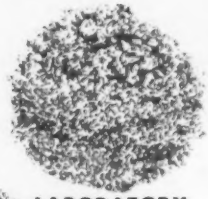

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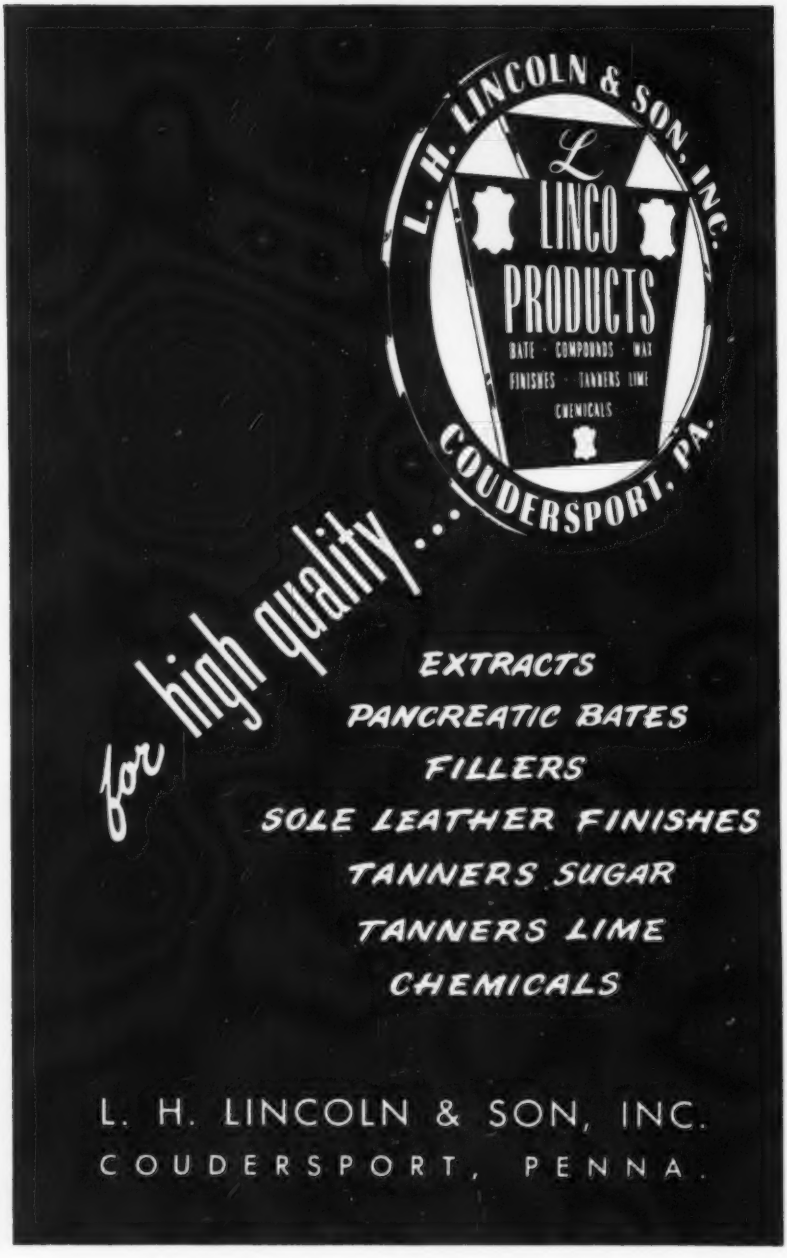
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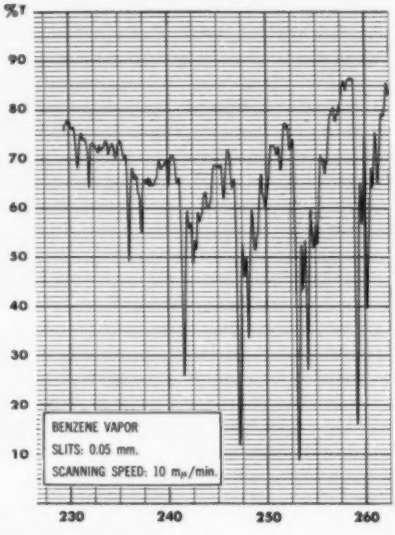

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RAPID MOISTURE DETERMINATION IN LEATHER

ROBERT STUBBINGS AND LOUISE AMBROSE*

Institute of Leather Technology
Milwaukee School of Engineering
Milwaukee 1, Wisconsin

ABSTRACT

A comparison of dielectric constant and resistance as a measure of the moisture content of leather has been made. The development of a d.c. resistance meter for leather moisture is described, and the operating parameters are evaluated. A commercial instrument for the rapid measurement of moisture content nondestructively is described covering the range of 9% to 40% moisture.

INTRODUCTION

The moisture content of leather is of great importance to the tanner, especially during the drying and conditioning operations. Present tanning methods for determining moisture involve cutting samples and determining the moisture in these samples by various oven methods. Usually the sampling is done in those areas of the side or skin which will have the least effect on the final leather, that is, around the periphery of the piece. Thus present methods are slow and destructive and do not tell the tanner about moisture conditions over most of the area of the side or skin tested.

ELECTRICAL METHODS

For many years moisture has been determined in many industries other than leather by various electrical methods. Most materials when they absorb moisture show large changes in two basic properties—electrical resistivity and dielectric constant. The changes in these properties have been the basis for the electrical determination of the moisture content. The electrical resistivity method has been applied to moisture determination in wood and wood products (1–3), plaster (4), paper and paper products (4), cereal grains (5), textiles (6), etc.

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The change in dielectric constant is made useful by measuring the high frequency capacitance (which is directly related to the dielectric constant), and this method has been applied commercially to grains and cereals (7–8), textiles (9), and other materials.

In 1949 Kremen (10) suggested the use of dielectric constant for the determination of moisture in leather. His data indicated that a high frequency capacitance measurement could be used for this purpose. Some time later the Matthews Moisture Meter (11) based on this principle was introduced commercially to the leather industry. Since capacitance is an inverse function of the distance between the plates of the capacitor, it was necessary that a correction for the thickness of the leather be made each time a capacitance measurement was made. This was accomplished by means of a low frequency induction coil which sensed the distance of the coil from an iron plate placed under the leather being tested. Thus the complete measurement entailed a capacitance measurement followed by a thickness measurement; then a calibrated correction for thickness was made and the moisture read from a chart or calibrated dial. The measurement required some fairly complex equipment, but it was rapid and nondestructive and could be applied over any part of the area of the side or skin desired.

LIFE LINES

DR. ROBERT STUBBINGS recently became associated with the Milwaukee School of Engineering where the Institute of Leather Technology was inaugurated after moving from Lehigh University. He is presently President-Elect of the ALCA. He received his education at Lehigh University where he earned his M.S. and Ph.D. degrees in chemistry while doing research under the late Dr. Edwin R. Theis.

MRS. LOUISE AMBROSE was with the Division of Leather Technology at Lehigh University for seven years where she worked on many phases of analytical chemistry of leather. She is presently on the staff of the Printing Ink Institute at Lehigh.

COMPARISON OF RESISTIVITY AND DIELECTRIC CONSTANT

In order to see whether resistivity or dielectric constant was the better property to test for moisture in leather, a manufacturer of resistive test equipment was asked to construct a meter designed for leather, and a Matthews Moisture Meter was obtained in the laboratory. The Kaydel Company (12) supplied a laboratory instrument designed to measure resistivity by comparison of the leather reading with a series of standard resistors. The measuring head consisted of two spring-loaded steel bars mounted parallel to each other, and the measurement was made by depressing the head against the leather surface until the head mounting came up against the leather.

A side of leather was cut into 150 4″-square pieces, and these were then all carefully equilibrated to a certain moisture level. The equilibration was done by moistening, then wrapping the pieces in Saran wrap in groups of 10, and allowing the packs to mull for 24 hours. After equilibration, each piece was weighed and immediately measured by both the Matthews and Kaydel machines. The necessary correction for thickness was made, and the Matthews readings were corrected for this. No correction was made to the Kaydel readings. The samples were then all rewet to a different moisture level, reequilibrated, and finally reweighed and measured by both Matthews and Kaydel devices. Six different moisture levels were used ranging from about 10% to about 30% moisture. After all the tests were completed, the samples were dried at 105° C. in a forced-draft oven for 24 hours and placed in desiccators. After cooling, they were weighed to determine their true dry weight. From these dry-weight values the true moisture contents of each piece at

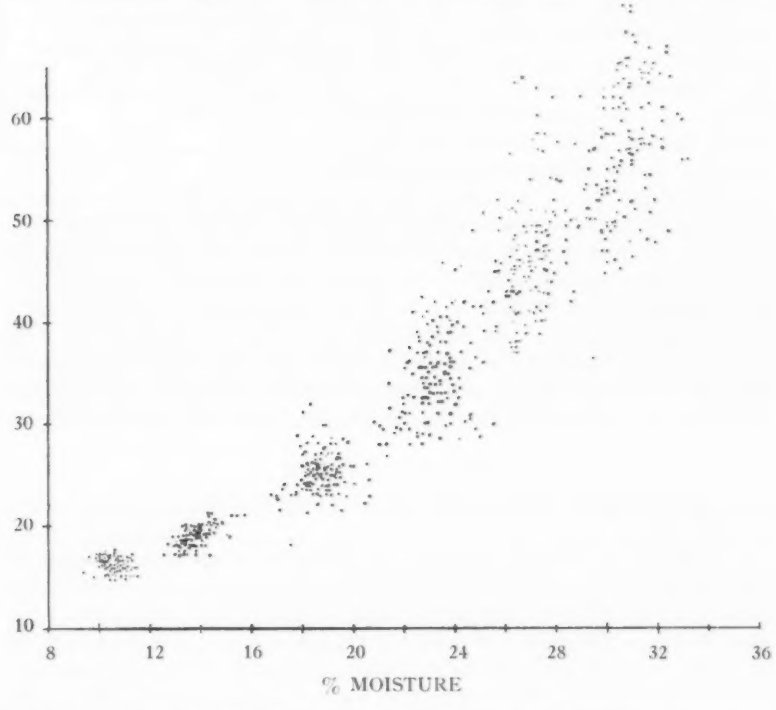

FIGURE 1.—Scale reading of Matthews Moisture Gauge (high-frequency capacitance-dielectric constant) versus true moisture (oven method) for 150 pieces of leather cut from same side after equilibration at various moisture levels. Each reading corrected for thickness.

each of the six moisture levels were calculated. These values were then plotted against both the Kaydel and Matthews scale readings. The results are shown in Figs. 1 and 2.

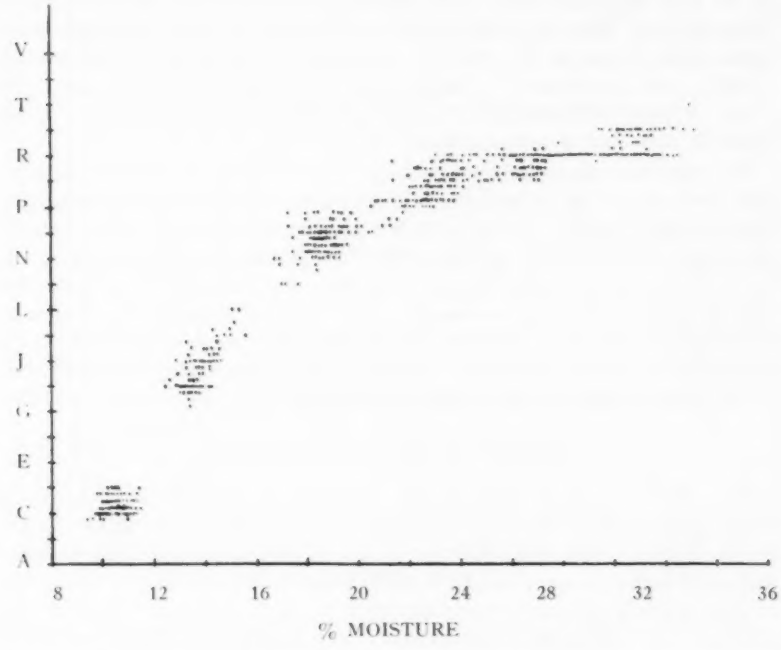

FIGURE 2.—Scale reading of Kaydel Moisture Gauge (d.c. resistance or conductivity) versus true moisture (oven method) for 150 pieces of leather cut from same side after equilibration at various moisture levels. No corrections for thickness made.

As can be seen, the dielectric constant method (Matthews) shows very large scatter at high moisture values and really becomes indeterminate at values above about 16% moisture. At low moisture levels the sensitivity by the Matthews device is relatively low (the curve is flat along the moisture axis), although useful measurements could probably be made up to about 16% moisture. The resistance method (Kaydel), on the other hand, shows high sensitivity at the low moisture levels—up to about 18%—and then shows a leveling off at values above about 22%. Because of the smaller spread of the data and the high sensitivity at the low moisture levels, it was decided to do further work using the resistance principle and to try to modify the apparatus to increase the sensitivity at the higher moisture levels.

DEVELOPMENT OF D. C. RESISTANCE MOISTURE METER

Through the cooperation of the manufacturer a modified d. c. resistance circuit was developed which impressed a much lower voltage on the leather at the high moisture levels, thus creating less electrochemical action during measurement. Also the general scale from 10–40% moisture was made much more linear based on the data for resistance versus moisture content obtained in the first series of measurements. In addition the meter was made direct reading with internval calibrating circuits so that moisture readings could be read directly on a calibrated meter face.

In order that measurements could be made rapidly on sides or skins in piles without having to remove the leather from the pile, a set of tongs was designed that could be slipped over the piece to be measured. The throat of the tongs was made deep enough so that any desired area of the piece could be reached with the leather flat on, or in, the pile. This first set of tongs was also designed so that measurements could be made between two electrodes on the grain side, two electrodes on the flesh side, or one electrode on the grain and one on the flesh side. A spring was mounted between the handles of the tongs to apply a relatively fixed pressure to the piece being measured.

EFFECT OF CONTACT PRESSURE

The effect of pressure between the electrodes was determined by making measurements at various moisture contents using increasing weight on the top electrode. It was found that the electrical resistance of the piece of leather decreased with increasing weight or pressure up to about 10 lb. and then leveled off to a fairly constant reading as shown in Fig. 3. Since the electrode size used was 3/8″ x 2″, this indicated that a pressure of at least 15 lb. per square inch should be used between the electrodes to achieve stable results. This effect of pressure between the electrodes is not primarily a thickness effect (that is, compression of the sample) but rather seems to be more closely connected with the establishment of proper contact between the surface of the electrodes and the leather. Thus it is important that the electrodes be parallel, that their faces be smooth, and that the pressure be reproducible from measurement to measurement.

EFFECT OF THICKNESS OF LEATHER

In preliminary studies with the first resistance meter it appeared that the thickness of the specimen had very little, if any, effect on the reading. This was carefully investigated with the new device, and the same general results were obtained. Two pieces of about 5-oz. side leather were carefully equilibrated in the same waterproof wrapper for 24 hours at about 25% moisture. Then each piece was measured in the tongs, and the two pieces were measured

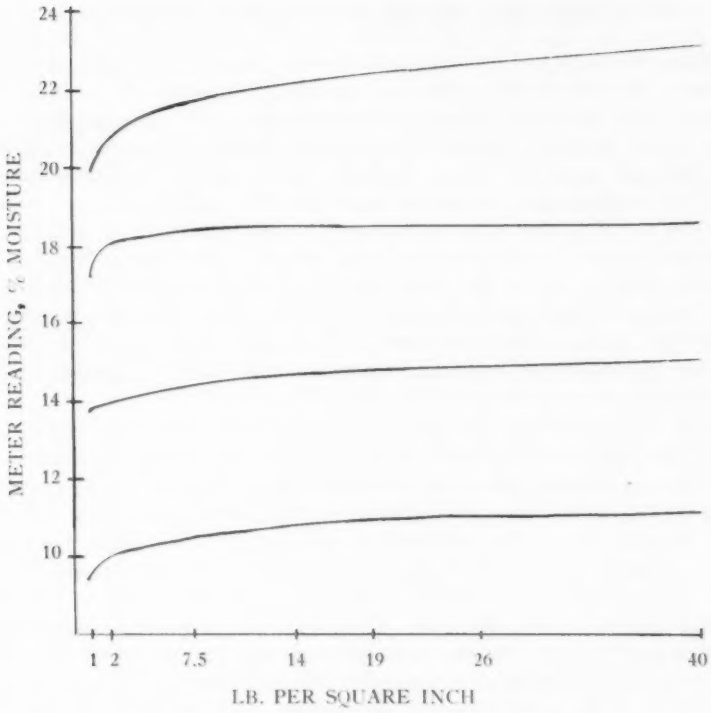

FIGURE 3.—Effect of pressure between electrodes on moisture readings (resistance gauge) for leather equilibrated to various moisture contents.

together in the tongs. The actual electrical resistance for the two pieces together was about twice the resistance of the average of the two pieces taken separately. Since the resistance versus moisture scale is more than *logarithmic* in resistance, this *linear* change in resistance with thickness has practically no effect on the moisture reading. To be more specific, the electrical resistance corresponding to about 25% moisture for a 5-oz. leather is 470,000 ohms, while the resistance corresponding to 23% moisture is 750,000 ohms. The change in resistance produced by doubling the thickness to 10-oz. leather at 25% moisture content would be to 940,000 ohms. This would read about 22.5% moisture on the meter. Thus, for practical purposes, the effect of thickness is small when moisture is being determined by the resistance method. On the other hand, doubling the thickness (without correction) using the capacitance method would give a value of about 12% moisture instead of 25%.

EFFECT OF MOISTURE DISTRIBUTION IN LEATHER

Using the tongs previously described (with 4 electrodes) it was possible to measure the rate of penetration and spreading of moisture in leather. This was done by measuring between the two electrodes on the grain side to get the "along the grain" result; by measuring between the two electrodes on the flesh side to get the "along the flesh" result; and by measuring between one flesh and one grain electrode to get the "through the leather" reading. For a piece of leather fully equilibrated (mulled overnight in Saran wrap) the three types of readings were almost identical, as would be expected. If a piece was wet up on the grain and flesh and immediately measured, the flesh and grain readings showed high moisture and the "through" reading low moisture, also as would be expected. Then, as the piece mulled in a waterproof wrapper the grain and flesh readings decreased and the "through" reading increased until at equilibrium all readings were equal.

Some very interesting new information came from this series of tests, however, in that the time required to reach equilibrium through the leather was less than 30 minutes, indicating that distribution of water through the thickness of the leather is a very rapid process. Why then do tanners invariably mull stock for staking for periods of about 24 hours? A very simple yet striking experiment gave us the explanation for this commercial practice. A 4″ x 4″ piece of leather was carefully moistened up to a line drawn on the piece on both the grain and flesh sides. The part of the piece on the other side of the line was left dry. The piece was then wrapped in Saran and mulled. After 24 hours the original dry area of the piece was still almost as dry as at the start of the experiment up to within ½″ of the line. Even after 1 week of mulling, equilibrium over the whole piece was not attained. Thus it is evident that moisture distributes laterally over the leather at a very slow rate, whereas it distributes vertically through the leather very rapidly. This result explains the long mulling periods used by tanners: they are trying to reach a more uniform moisture condition over the whole area, and this takes considerable time. It further points the way toward more efficient rewetting methods prior to mulling. The important objective of the rewetting should be the even distribution of the water over the area of the piece, rather than a certain level of total pickup by the piece. Since the grain after pasting and even after paste washing is relatively nonuniform in wettability, it is suggested that carefully controlled spraying of the flesh side may produce a much more uniform moisture distribution over the area of the side or skin.

The rapid distribution of moisture through the skin indicated to us that measurements both across the surface and through the piece would be impractical for production use, and further that either a through measurement or a surface measurement should be satisfactory on a piece at equilibrium. We feel that a set of tongs measuring through the leather is the best practical

answer for measuring moisture in a pile of leather at staking but that a set of surface electrodes would be very useful for checking moisture on the pasting plates, for example.

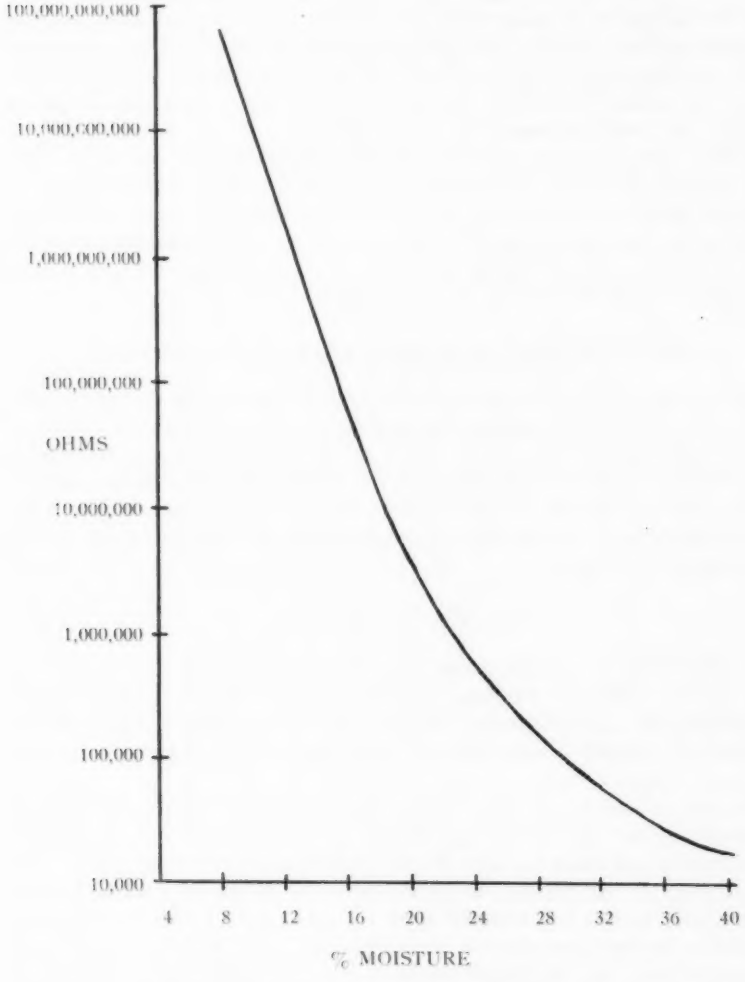

FIGURE 4.—Electrical resistivity of leather in range of moisture content from 10% to 40%. Five-oz. leather; ¾ sq. in. electrode area; 10 lb. pressure on electrodes.

CALIBRATION OF ELECTRICAL RESISTANCE VS. MOISTURE

The information about the very slow distribution of water laterally through the leather also led us to a further precaution in the calibration of the electrical resistance scale by means of the oven method. It is absolutely essential that the calibration be done on a piece that is at complete equilibrium over its entire surface; otherwise the oven reading for moisture will not correspond to the reading taken at one small place on the piece with the tongs. To calibrate the resistance scale to the true moisture scale, we carefully spread moisture as evenly as possible over the leather piece with a paint roller on both flesh and grain sides and then mulled for 24 hours in Saran wrap. We then weighed the piece and rapidly took several readings with the tongs in different spots on the leather. Later the piece was oven-dried, desiccated, and reweighed in order to calculate the true or oven-method moisture content. The curve for moisture content versus resistance is shown in Fig. 4 for the electrical measurement circuit later described.

EFFECT OF TYPE OR KIND OF LEATHER ON READING

Seven different leathers were equilibrated to various moisture contents and read on the device with the results shown in Fig. 5. With the exception of the stuffed leather they all fall along the same general curve within about $\pm 2\%$ from the curve. These data indicate that one general scale for moisture versus resistance should be useful for tannery measurements and that if for some reason precise results were needed, a calibration could be carried out on the leather in question.

ELECTRONIC CIRCUITS

In order to have a useful instrument for rapid measurements in the tannery, it was decided to try to linearize the resistance versus moisture function and at the same time to spread it out over four independent scales for easier reading. In addition it was decided to make the instrument portable for best use in the plant.

The circuit used for the three highest resistance (lowest moisture) scales is shown in Fig. 6. Adjustment circuits are provided to take care of aging of the batteries and tubes in order to get the most useful life from both. The linearization of the function is accomplished by means of a diode (1N34) shunt. All switches and terminal posts are constructed with extremely high insulation materials to prevent shorting when measuring the very high resistance values on the lowest moisture scale. Approximately 50 volts is impressed on the leather sample in these three lower moisture measurements.

The circuit used for the high moisture (27–40%) scale is shown in Fig. 7. In order to reduce polarization and electrochemical effects a much lower

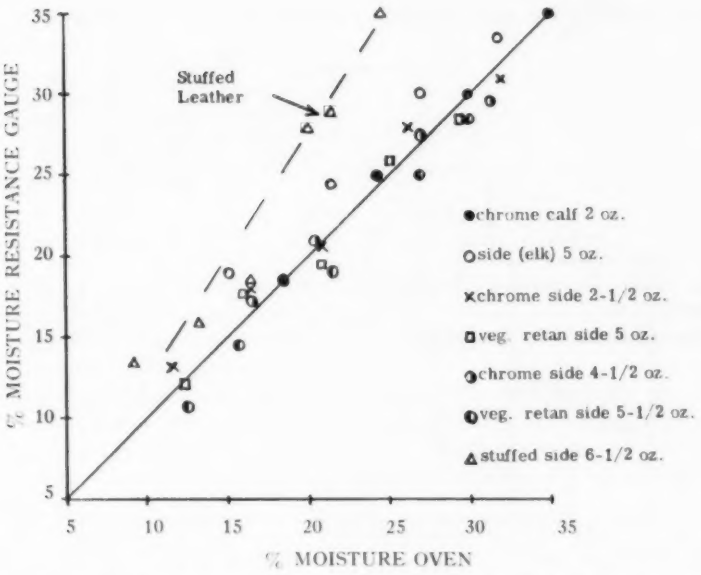

FIGURE 5.—Effect of type of leather on moisture reading using resistance gauge.

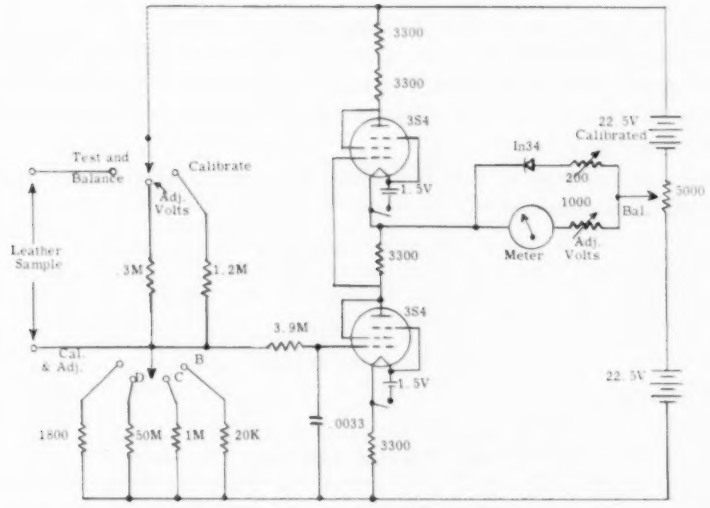

FIGURE 6.—Circuit used to measure moisture content of leather from 9% to 27% moisture. Scale D = 9–13%, Scale C = 14–19%, Scale B = 19–27%.

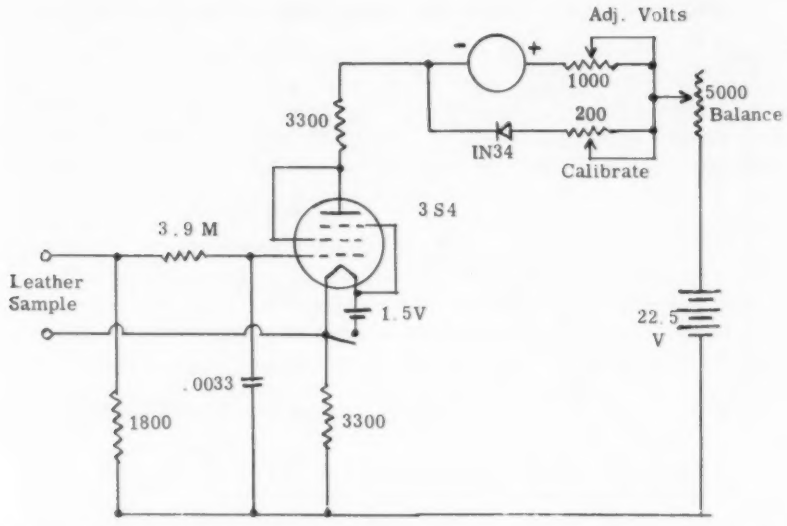

FIGURE 7.—Circuit used to measure moisture content of leather from 27% to 40% moisture. Notice voltage applied to leather is due entirely to small voltage developed across 3300-ohm cathode resistor.

potential is applied to the leather. The actual potential applied is the drop across the 3300-ohm cathode resistor, which at full scale on the meter amounts to less than 1 volt. It is this modification which made it possible to operate up to moisture levels of about 40% instead of the leveling off at about 28%, as shown in Fig. 4.

These circuits then provide four scales of moisture on the same meter face ranging as shown below:

Scale A 27–40%
" B 19–27%
" C 14–19%
" D 9–13%.

USE IN THE TANNERY

The commercial instrument, as finally developed, has been tested in tannery use for various purposes and found to give useful results in the following

operations for both average moisture and distribution of moisture over the side or skin:

1. after paste drying
2. before staking after mulling
3. after toggling
4. after sawdusting
5. at buffing
6. at finishing

The instrument *cannot* be used to determine moisture of leather at any part of the wet processing, however. When the leather is wet, the conductivity is dependent upon factors such as ionic strength, acidity, temperature, etc., and is not related to moisture content.

The scales of the commercial instrument are calibrated for an average leather, and if precise moisture results for a given type of leather were required, a separate calibration would have to be carried out in the tannery. This should be done on pieces that are uniformly equilibrated as described previously.

At present the commercial instrument is being manufactured by Amtronics Inc., 1140 8th Avenue, Bethlehem, Pennsylvania.

ACKNOWLEDGMENT

The authors wish to thank the S. B. Foot Tanning Co., A. F. Gallun and Sons Corp., Ohio Leather Co., Pfister and Vogel Tanning Co., Fred Rueping Leather Co., and Albert Trostel and Sons Co. for their encouragement of, and contribution toward, this work and for the assistance of their personnel in running tannery trials of the instrument.

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Received December 27, 1960.

DISCUSSION

DR. SEYMOUR S. KREMEN: I think we all ought to congratulate Dr. Stubbings for a splendid advance in instrumentation, particularly the use of the relatively low current method of measuring the resistance of the wet leather to avoid the problems caused by the high-current carrier in capacitor measurement or resistance measurement where you use a relatively large voltage. I think it is high time we tried to get tannery procedures on a basis of quantitative control rather than human experience and intuition.

I have two questions. One is about the question of contact resistance, where, in the resistance measurement, you say the thickness is not a terribly important factor in the calibration. In the capacitors' measurement it necessarily is. The thickness of the dielectric is right in the capacitors' formula. But contact resistance, depending upon the amount of moisture present and the type of leather, can be fairly important. I was wondering how you managed to solve that.

The other question is about the reproducibility of a single measurement. If you make the measurement at the same place within a short time and repeat it several times, what kind of variation would you expect to obtain?

DR. STUBBINGS: Well, in answer to the first question, there are really two problems involved. Contact resistance I do not believe is the major problem if, by contact resistance, we mean the effect at the inner face of the electrode on the leather, although that may be the fundamental one.

What it resolves itself into is a pressure problem. The result will change depending upon the pressure with which the electrodes are applied to the sample. And the only solution we have for this is to provide a spring in the back of the tongs which applies approximately the same pressure to the sample each time. This is certainly not a good or quantitative proposition, but it is reproducible within a set of tongs. And since normally calibrations would be done with instruments in use, I think that it is probably adequate for tannery work.

However, I don't understand precisely what this pressure thing is. It may be a contact potential proposition, or it may be an actual moving of the fibers and thus a change in the mat which the electrodes are measuring.

As to the reproducibility of a measurement, if we reproduce a single measurement on a single piece which is then equilibrated, then the reproducibility on the face is less than 0.1% of moisture—provided someone does not change the pressure on the tongs. You can observe this for yourself. I brought some wet samples of different types of leather along with me, and you can see that if you put them in the tongs and let the tongs apply the pressure, you will get one result; while if you put them in the tongs and squeeze the tongs, you will get another result. So in this respect if anyone could devise a more reli-

able way of applying the same pressure all the time to the sample in a simple device, it would be a great improvement. It is about the order of 0.5–1% if you squeeze the tongs.

PRESIDENT MAESER: Is there a drift observable?

DR. STUBBINGS: There is no drift in the low moistures ranging up to 27%. In the high moisture ranges there is an observable drift if you leave it over a long period of time. Over a two- or three-minute period it is not at all large.

DR. ERNEST R. STEIN (Whitehall Leather Co.): Does the amount of fat, and do different types of fat, interfere with the measurement of moisture?

DR. STUBBINGS: For regular fatliquored production leathers we have seen no large differences. Stuffed leathers, however, do show a distinctly different curve from the standard fatliquored leathers.

DR. BUECHLER (Rohm & Haas Co.): Do you find differences depending on the chrome content of the leather?

DR. STUBBINGS: We don't know. The leathers we have tested do vary in chrome content but not over an extremely wide range. They vary maybe in the order of 2–3.5%. If we went over, say, a 1–10% range, we might see a difference.

However, we must remember that what we are talking about is leathers that have already been washed after the final fatliquoring and dried on the pasting plates, or tunnel-dried, and which do not contain a large quantity of ionic salts such as wet leather would in processing. This is not applicable in any sense to wrung-out blue stock or anything of that sort, nor is it applicable to a cured stock with a high salt content. It will not work for that. It will only work on leather after it has been essentially washed fairly free of salts.

DR. KREMEN: Have you, or has anybody else, tried it for a normally loaded salt leather?

DR. STUBBINGS: No, we have not. I don't know what would happen.

RICHARD N. JONES (A. C. Lawrence Leather Co.): Is this instrument commercially available, and if so, what is the price on it?

DR. STUBBINGS: Well, this particular instrument was built, as I say, by this company specifically for this project and for this purpose. At the time it was built we had six built for tannery work. The price on the instruments

at that time from the company was of the order of $400. This does not include the tongs that are with it. These were made locally in our area.

I cannot guess what a final production model would cost. I would feel that if it were going to be produced commercially, certain modifications should be made to it. And whether or not they are willing to produce it at a price that will meet the tanner's needs, I do not know. I am trying to find this out right now.

DR. L. SELIGSBERGER (Quartermaster R & D Command): I wonder whether such an instrument can be developed to measure the moisture on one side of the leather—whether you absolutely need tongs?

DR. STUBBINGS: We have done this. Our first instruments were built with paired electrodes—with two electrodes in each head, independently coming back to a switch so that we could measure moisture across a surface or through the leather. This was an interesting instrument, since it gave us some information about the difference of rate of penetration of moisture in the leather.

Our conclusion, however, was that in normal practice this was not as reliable as measurement through. Consequently, our final design employs measurement through the leather with the electrodes. However, you can measure on the surface, and as far as I can see, if you are willing to calibrate an instrument based on this principle, it ought to work. The difficulty would be that you would have to know, just as you do with this, the conditions of equilibration of the sample. If a sample were wet, let us say to 28% moisture, and then allowed to air-dry from the surface, you would begin to get entirely different results with the surface electrodes measuring the moisture than with the electrodes through the leather. These provide some interesting information, as a matter of fact, in terms of rates of drying.

GEORGE A. BUTZ (Endicott-Johnson Corp.): I would like the maximum and minimum that you could use this for. I realize that there are problems in the wet processes, but I am thinking of applications like mulling in shoe factories. How high can you go, and how low, and still be within 1% of moisture or, say, 2%?

DR. STUBBINGS: If we say calibrated to the particular leather, then we can say 1%. Now, we can go from 9% moisture to about 35% moisture. You cannot go higher.

MR. BUTZ: You cannot go higher?

DR. STUBBINGS: I do not believe so. Something happens above 35% which is not the same thing. Above 35% you have free water in the leather. It is

not bound to the fiber, and the results then become a flat line in terms of conductivity because you are simply measuring the conductivity of the salts dissolved in that water, and that is not what you are trying to do.

MR. BUTZ: And you cannot go below 9% for the same reason?

DR. STUBBINGS: This is true of practically any instrument. If you go below 9%, it will require specialized conditions of measurement. It is not the problem of the instrument but the problem of the air and conditions around the instrument. You see, 9% moisture is approximately a resistivity of 500,000,000 ohms. As soon as you get up in ranges like this and above, moisture in the air will give you a leakage path that is greater than the leakage path through the leather. So you would have to do this under peculiar circumstances to make it work.

DR. KREMEN: Apropos of Dr. Seligsberger's question, if I remember that meter, it had a leather bedplate.

DR. STUBBINGS: The original meter we actually used was a probe that went down on the metal plate, the metal plate being one electrode and the probe containing the other electrode. Any method of establishing an electrical path across the leather or through the leather is readable by this type of measurement. The conditions of the probe are more or less up to the imagination of the user.

One particular application that has been made of this, that I know is in use in production, is the measurement of the moisture during mulling in a shoe factory, where they need a very rapid measurement of the mulling moisture. And this has been used with modified probes to do the job. The electrodes fit a very small sample and are applied from one side only.

THE INFLUENCE OF BACTERIA ON DEPILATION OF HIDES BY ENZYMES*

THEONE C. CORDON, A. L. EVERETT, AND W. WINDUS†

Eastern Regional Research Laboratory‡
Philadelphia 18, Pennsylvania

ABSTRACT

Hides and skins as received in a tannery are contaminated with bacteria, and the reduction in salt content after soaking makes them susceptible to degradation. The conditions used for enzyme unhairing also favor the growth of bacteria, a factor ignored by some investigators.

Of a number of disinfectants studied, several were found to control bacterial growth at concentrations which were not harmful to the hair-loosening enzymes.

Although the unhairing solutions were not sterilized, there was no apparent damage to the hide, no putrid odor, and no change in the unhairing action, if the bacterial population was kept under about 100 million per ml.

INTRODUCTION

Although the use of enzymes for loosening the hair on hides and skins has been advocated for at least 50 years (1, 2), little attention has been paid to the possible role of the microorganisms which develop in the liquors. Although disinfectants have been and are used routinely in the commercial application of enzymes for unhairing (2, 3), it seems very likely that many of the results reported were affected by the bacteria present in the unhairing medium. Many workers fail even to mention bacteria, and some (4) have expressed the belief that the results of others were influenced by bacterial contamination.

Numerous workers have studied the "abnormal" bacteriology of hides to seek the causes of various troublesome defects. Others have indicated the large variety of organisms to be found on salted hides and skins. Anderson

*Presented at the 59th general meeting of the Society of American Bacteriologists, St. Louis, Missouri, May 10–14, 1959.

†Biographies of the authors may be found in the February *Journal*.

‡Eastern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

The mention of commercial items or companies in this publication does not constitute an endorsement by the Department of Agriculture over others not mentioned.

(5) was more concerned with the so-called normal flora; he tested a number of proteolytic strains in pure culture but found that only the aerobic spore-formers could digest hide substance, which we (6) have also confirmed. However, he made the interesting observation that most of the strains tested caused complete loosening of the hair (after 3 days at 37°C.). Since any organisms originally present could readily be carried over with the hides, it is important that they be controlled in the enzyme liquors.

Early in our studies of enzyme unhairing (7) a preliminary investigation of some suitable disinfectants was made, and as a result, phenylmercuric acetate was routinely used in our unhairing solutions. Later we included BSM-11, which also contains phenylmercuric acetate. Shuttleworth (8) had recommended zinc or mercuric chloride, among other materials, from a comparative study of soak disinfectants. In a recent study of bactericides for use in sheepskin soaks, Richardson (9) tested a number of materials by a laboratory screening method. Phenylmercuric chloride and several silicofluorides, among others, were found to be effective, while 2-naphthol was not. He noted that bacterial counts ranged from 100 million to 100 billion per ml. in soak waters from packs showing damaged pelts.

When larger-scale tests were started, it was found that if hides were soaked with inadequate amounts of disinfectant, the bacterial population built up to the point where the disinfectants we were using could not control their growth during the enzyme treatment. Under these conditions extremely putrid odors often developed, and extensive hide damage sometimes occurred. Tancous (10) has found a *Clostridium sp.* in salt-cured hides which produces a potent collagenase. This or other collagenase-producing bacteria were no doubt responsible for the hide damage. With proper soaking conditions the disinfectants we used controlled growth satisfactorily.

In an effort to throw light on the problem of inconsistency of hair-loosening results a study was made of the bacterial populations of a large number of enzyme unhairing liquors. The results are reported here.

MATERIALS AND METHODS

Enzymes.—The enzymes used were mostly commercial products. They are listed with their biological sources and suppliers in Table I.

Antiseptics.—The antiseptics, their composition and suppliers, are shown in Table II.

Hides.—The hides were obtained from the M. A. Delph Company, Indianapolis, Indiana. Immediately after flaying they had been fleshed, demanured, and brine-cured. Before use they were washed in a large drum for 1–2 hours and drained.

TABLE I

ENZYMES USED

Enzyme	Source	Supplier
Protease 15 Concentrate	Bacterium	Rohm & Haas Philadelphia, Pa.
Protease 4511-3	Bacterium	Wallerstein Co. New York, N. Y.
Papain	Papaya	Nutritional Biochemicals Cleveland, Ohio
Keratinase	Streptomyces	Institute of Microbiology New Brunswick, N. J.
Viokase	Pancreas	Viobin Corporation Monticello, Ill.
Protease L 56-D L 306, L 305	Bacterium	Pabst Laboratories Milwaukee, Wisconsin
HT Proteolytic, 3, 110 416, 19-20	Bacterium	Takamine Laboratory Clifton, N. J.
HT Concentrate 4903	Bacterium	Takamine Laboratory Clifton, N. J.
Bromelin	Pineapple	Takamine Laboratory Clifton, N. J.

TABLE II

DISINFECTANTS USED

Name	Composition	Supplier
Mersolite-8	Phenylmercuric acetate (PMA) 97.5%	F. W. Berk, Inc. Wood-Ridge, N. J.
BSM-11	10% Phenylmercuric acetate, 50% potassium 2, 4, 6-trichlorophenate, undisclosed solvents	Buckman Laboratories Memphis, Tenn.
Dowicide B	Sodium 2,4,5-trichlorophenate	Dow Chemical Company Midland, Michigan
Sterozol S	Pine oil, cresols orthophenyl phenol, penta-chlorophenol, 2-naphthol	Wallerstein Co. New York, N. Y.
Sodium dithionite	$Na_2S_2O_4$	Eastman Organic Chemicals Rochester, N. Y.
2-Naphthol	2-naphthol	J. T. Baker Chemical Co. Phillipsburg, N. J.

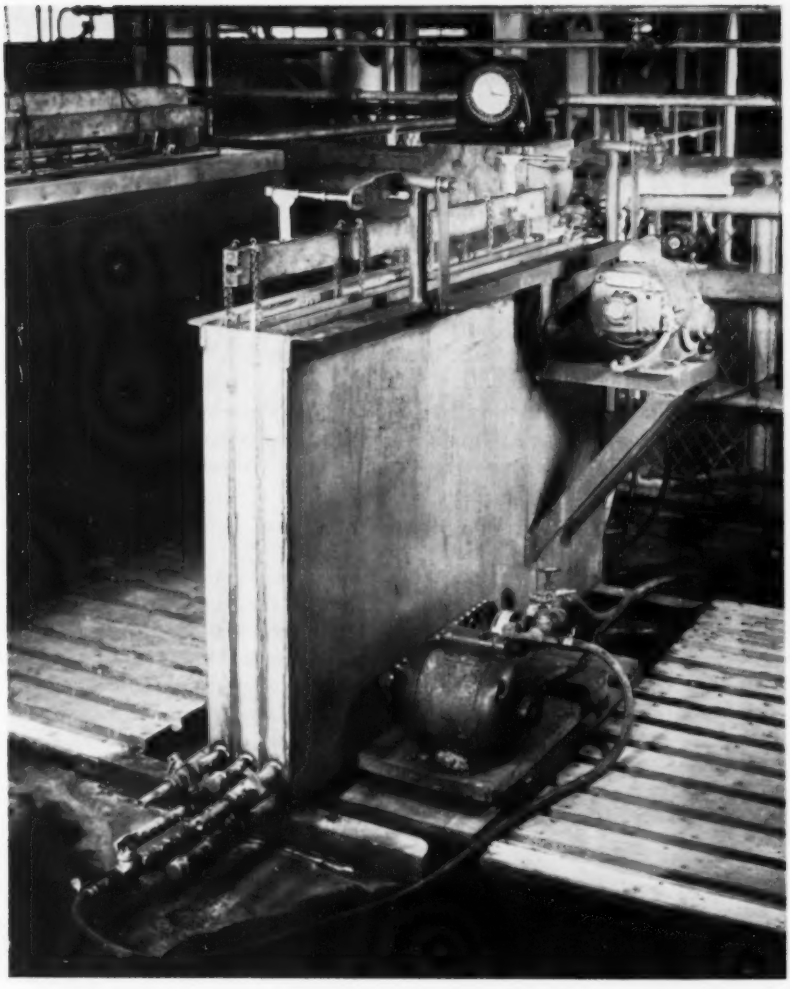

FIGURE 1.—Stainless steel unhairing vat. See text for description.

Unhairing procedure.—The enzyme and additive material under test were dissolved in water and placed in either of the two outer sections of a stainless steel rocker unhairing vat shown in Fig. 1. The center section of the unit contained water and served as a temperature control bath. The water was continuously circulated by pumping out at the top and in at the bottom.

A 1000-watt heater in the bath, connected through a thermoregulator, maintained the temperature at the desired point. Hide pieces consisting of bend and belly or shoulder portions, weighing about 6 to 8 kg., were suspended in the vats. Stainless steel rods were tied to the lower edges of the hides to keep them vertical during rocking. The vats contained 35 l. of solution, making the hide-to-solution ratio about 1 to 5. The hides were rocked at least part of the time they were in the solution, at the rate of 50 cycles per minute with a 4½″ stroke. After 20–24 hr. at 30°C. hair looseness was judged by the thumb test and by unhairing with a beam knife. After unhairing, the hides were examined for defects.

Estimation of numbers of bacteria.—Plate counts were made by a standard procedure using Bacto nutrient broth containing 1% NaCl, 0.3% glucose, 0.1% Bacto yeast extract, and 2% Bacto agar. Appropriate dilutions of samples were made in sterile 1% saline. When phenylmercuric acetate was present in the enzyme solution, 0.1% sodium thioglycollate was added to the dilution blanks to counteract this disinfectant carried over in the inoculum.

EXPERIMENTAL

The bacterial population of an enzyme unhairing liquor would be the result of the growth of organisms added with the hide, the water, and the enzyme. Table III shows the numbers of viable bacteria present in some of the enzyme preparations. It is evident that viable bacteria would supply a heavy inoculum to the unhairing liquors. The cells present in the preparations containing a high number of bacteria were found to be mostly in the spore form. Under

TABLE III

NUMBERS OF VIABLE BACTERIA IN ENZYME PREPARATIONS

Enzyme	Plate Count (Air-Dry) millions/g.	Enzyme	Plate Count (Air-Dry) millions/g.
Protease L306	880	Viokase	<0.01
Protease L305*	180	Bromelin	0.09
Protease L 56-D	330	Protease 15 Concentrate	11
HT Proteolytic 110	27		
HT Proteolytic 3	120	Protease 4511-3	0.04
HT Proteolytic 416	217	Papain	<0.01
HT Proteolytic 19-20	16	Keratinase*	4.9
HT Concentrate 4903	50		

*Liquid preparation, count per ml.

the conditions used for unhairing, these spores germinated in a few hours and built up to about 300 million bacteria per ml. within 24 hr. The question naturally arose as to whether these bacteria, which were presumably the same culture which produced the enzyme, would contribute to the depilatory action of the liquor. Several laboratory tests were run with Seitz-filtered HT Proteolytic 3 enzyme. Typical results are reported in Table IV. It is

TABLE IV

THE EFFECT OF REMOVING BACTERIA ON THE HAIR-LOOSENING ACTIVITY OF HT PROTEOLYTIC

Treatment	PMA %	Zero Time	20-hr. Incubation	
		Bacterial Count No/ml	Bacterial Count No/ml	Hair Loosening
None	None	276,000	400,000,000	Very good
None	0.045	106,000	3,870,000*	Very good
Seitz-filtered	0.045	70	3,000,000*	Very good

*Most of these colonies were a *Pseudomonas sp.* which proved, on isolation, to be relatively resistant to PMA but did not affect hair loosening.

clear that filtering out the viable cells did not decrease the unhairing action of the enzyme preparation; nor did allowing them to develop increase it. The fairly high counts which developed in the solutions containing phenylmercuric acetate were due to a *Pseudomonas sp.* originating from the hide. This organism appeared fairly frequently in the unhairing liquors. It was isolated and found to be about six times more resistant to the disinfectants than the remainder of the population. It produced a characteristic aromatic odor but did not exert any hair-loosening action when hide was placed in a whole broth culture. Furthermore, it did not produce any detectable effect when present in the enzyme unhairing solution.

The relative effectiveness of several disinfectants was demonstrated in the following tests: Hides were placed in 35 l. of 0.1% HT Proteolytic 110 solution in the unhairing vat. The disinfectants and their concentrations as percent in solution are shown in the graph (Fig. 2), plotted against the corresponding bacterial count at the end of each test. After 21 to 23 hr. of continuous rocking at 30°C., the hides were removed and unhaired on a beam. The ease of unhairing was rated as very good (VG), good (G), or fair (F) and is shown on the graph. After the loss in volume (about 3 l.) was restored with fresh solution of the same composition, other hide pieces were then treated with the same liquor and evaluated as before. Note that all the counts remained within the 100 million range.

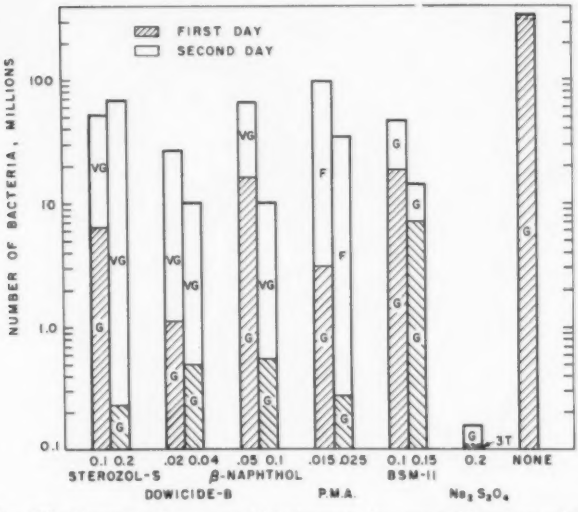

FIGURE 2.—Effectiveness of several disinfectants for controlling bacterial growth in enzyme unhairing solutions. Concentration given is percent in solution. The letters in the bars refer to ease of unhairing. VG = very good, G = good, and F = fair. The cross-hatched sections of the bars show the bacterial count after one use (24 hr.). The open sections show the counts after a second hide had been incubated a further 24 hr.

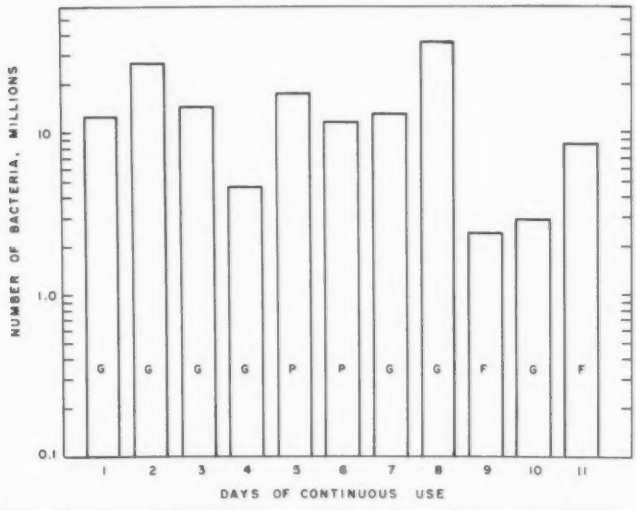

FIGURE 3.—Numbers of bacteria in a continuously used enzyme unhairing solution. The enzyme was HT Proteolytic 0.1%, and the disinfectant BSM-11, 0.1%. The letters in the bars refer to ease of unhairing. G = good, F = fair, and P = poor.

In another experiment an enzyme unhairing solution (0.1% HT Proteolytic 110) was preserved with 0.1% BSM-11 and reused daily for 11 consecutive days. Solution lost by removal of the hide was replaced each day with fresh solution before adding another hide. The results of tests for hair loosening and bacterial numbers are given in Fig. 3. In this series the counts were controlled within a range of 40 million, despite the frequent introduction of more organisms and nutrients.

DISCUSSION OF RESULTS

The results given in Table III show that the bacterial counts of enzyme preparations vary widely. As would be expected, the bacterial enzymes contained the highest numbers of viable cells. They varied between 11 and 880 million per gram of air-dry material and would obviously produce higher initial counts in the unhairing solutions.

The numbers of bacteria developing in such liquors can be quite easily controlled by the use of common inhibitors as shown in Figs. 2 and 3. BSM-11 at 0.1% concentration held the population below 40 million per ml. for 11 days of continuous use. All the disinfectants tested held the count below 100 million per ml. for two days, whereas without disinfectant the count reached 325 million in 24 hr. No evidence of the enhancement of hair-loosening action by bacteria present in the unhairing solutions could be detected. Although some of the results given in Fig. 2 seem to indicate an increase in hair loosening with increasing bacterial numbers, this is probably a coincidence due to hide variation. Data from eight other experiments, with widely varying degrees of control, do not show any correlation of hair loosening with bacterial numbers (Table V).

Under the conditions of these tests, no damage to the hides could be detected even when the bacterial count reached 325 million per ml. of solution, as was the case in the control.

The lack of uniformly good unhairing in the test reported in Fig. 3 is probably due to the low concentration of enzyme used. One-tenth percent of this enzyme is a border-line concentration. Some hides unhair satisfactorily at this level, and some do not. If sufficient enzyme had been used (0.15% to 0.2%), it is believed that the unhairing would have been satisfactory. Our primary objective was to detect any differences due to the bacteria present, rather than to produce consistently good unhairing.

As pointed out earlier, it is well known that if bacteria are allowed to grow unchecked, they may damage hide substance. These results indicate, however, that if reasonable precautions are taken to keep a large population from developing at any step in the processing, no trouble should be experienced with bacteria in a short enzyme unhairing process.

TABLE V

RELATION OF BACTERIAL COUNTS IN ENZYME LIQUORS TO HAIR LOOSENING

Enzyme Liquor		
Times Used	Bacterial Count Thousands/ml	Hair Looseness
1	26	Fair
2	4,400	Fair
1	0.2	Good
2	24,000	Fair
1	55	Good
2	18,000	Good
3	34,000	Good
1	12,600	Good
2	1,410	Good
3	47,000	Good
4	160	Good
1	351,000	Good
2	320,000	Good
1	24.7	Good
2	156	Good
3	115,000	Fair
4	283,000	Fair to poor
1	4,700	Good
2	33,700	Good
3	28,000	Good
1	——	Good
2	7,200	Very good
3	11.9	Very good

It is interesting, and possibly commercially significant, that an enzyme solution could be reused so many times. It appears that if a higher concentration of enzyme had been used in our test, good hair loosening would have been maintained throughout the entire period.

SUMMARY

If bacteria are allowed to grow unchecked in enzyme unhairing liquors, the hides may be damaged severely. Common disinfectants in moderate amounts will control the growth. If the bacterial numbers are held within about 100 million per ml., the hide substance is not digested. In these tests large numbers of bacteria in the unhairing solution did not increase the hair-loosening action.

ACKNOWLEDGMENT

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The second of the ethical values of scholarship is the obligation to publish. Again, the propriety of this obligation is seldom called into question. But carrying it out often involves the scholar in complicated casuistical problems. When, for example, should he publish? We all have among our friends scholars who publish at the drop of a hat—and what they publish often has about the same global significance. Others accumulate, revise, supplement, delete, emend—and the advancement of knowledge is held back by the perfectionism of the scholars. In what style shall a scholar write? Shall it be one which will be sufficiently technical and refined to satisfy other scholars but leave the general reader confused and frustrated, or one which will communicate to the common man but will evoke from the author's colleagues bitter charges that he is sacrificing scholarship for royalties? When shall a scholar acknowledge sources? The answer is, always. And the ethical issue seems quite clear. Indeed, the violation of this principle is perhaps more heinous than that of tearing pages from a library book. The student who submits a term paper consisting of extracts from published works without benefit of quotation marks and footnotes has committed a truly dastardly crime, and a scholar who does the same thing and is caught by the copyright laws before he is caught by his conscience is simply not a scholar. I might add, in view of recent events, that a scholar who participates in a rigged quiz program and confesses only when he is caught by a Congressional investigating committee should hardly be surprised if his actions produce a feeling of nausea in scholars and moralists alike. But how hard it often is to know one's sources. Are they simply what one is reading when he gets an idea? And is the idea in the text or is it merely suggested by the text? And suppose one gets a new idea every time he rereads a book, as might well be the case if he is reading Plato or Emerson or Carlyle. Does scholarship which adheres to this principle then degenerate into an autobiographical account of one's intellectual adventures in a library? Must a scholar who writes a book on geometry make due acknowledgment to Euclid, and must a scholar who writes a book on atomic physics always cite Democritus?

—A. Cornelius Benjamin, "The Ethics of Scholarship", *The Journal of Higher Education*, Vol. XXXI, Dec., 1960, pp. 479–80.

ALCA NEWS

Proceedings, Reports, Notices, and News

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Asociación Argentina de los Químicos y Técnicos de la Industria del Cuero

We are glad to announce the formation of the Asociación Argentina de los Químicos y Técnicos de la Industria del Cuero by the leather chemists and tanners of Argentina. Their address is Calle Belgrano 3978, Buenos Aires. They have also begun the publication of a journal which is available for the equivalent of 150 Argentine pesos a year. We are glad to agree to their suggestion that we exchange journals.

Dr. Ernesto Mezei, the author of a book on quebracho, is president of the Association. Dr. Carlos Durlach is vice-president; Dr. Jorge R. Camilli, secretary; Ing. Carlos Bühler, prosecretary, and Ing. Francisco Grünwald, treasurer.

Their association is a member of the International Union of Leather Chemists Societies. An invitation has been extended to them to take part in the meeting of the International Union in Washington, D. C., August 20 to 25.

We congratulate them upon the formation of their association and wish them every success in the application of chemistry to the production of leather.

PROCEEDINGS OF 1957 I.U.L.C.S. SYMPOSIUM

The Italian Association, in conjunction with the Leather Experimental Station of Naples, has printed a booklet containing the proceedings of the Symposium in Leather Manufacture which was held during the 5th Conference of the I.U.L.C.S. in Rome, 1957. Each discussion is in the language of the speaker.

This booklet is obtainable from the

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ALCA-ASTM JOINT COMMITTEE ON LEATHER

December 8–9, 1961
Sheraton Atlantic Hotel
New York, N. Y.

The subcommittee and section meetings began at 10 A.M., December 8, and continued until 3 P.M., December 9. A list of those in attendance and the detailed minutes follow.

MILTON BAILEY
Secretary

Attendance List

Joseph R. Kanagy	National Bureau of Standards
William N. Wacker	John R. Evans & Co.
Joseph Naghski	Eastern Utilization Res. & Dev. Div.
Edward T. Steiner	Quartermaster R. &. E. Center
Charles F. Dudley	Dow Corning Corp.
Robert Shaw	Rohm & Haas Co.
C. Paul McKee	International Shoe Co.
M. H. Olson	Minnesota Mining & Mfg. Co.
N. Clifford Benrud	S. B. Foot Tanning Co.
M. M. Baldwin	Battelle Memorial Institute
E. M. Filachione	Eastern Utilization Res. & Dev. Div.
F. P. Luvisi	Eastern Utilization Res. & Dev. Div.
M. Bailey	U. S. Navy Research & Development Facility
R. M. Lollar	Armour Leather Co.
G. L. Leithauser	General Motors Corp.
J. W. Zimmerman	General Motors Res. Div.
Lee P. Witnauer	Eastern Utilization Res. & Dev. Div.
B. L. Lewis	Olson Testing Machine Co.
Samuel F. Etris	ASTM
R. L. Young	International Shoe Co.
G. H. von Fuchs	Consultant
Charles W. Mann	Lowell Technological Institute Research Foundation
R. G. Ashcraft	Endicott Johnson Corp.
Robert Stubbings	The Milwaukee School of Engineering
William T. Roddy	Tanners' Council Research Laboratory
Shu-Tung Tu	United Shoe Machinery Corp.
H. Haemisegger	Newark Leather Finish Co.

ADVISORY COMMITTEE

The Advisory Committee met at 11 A.M., December 9, 1960. The following members were present: R. G. Ashcraft, M. Bailey, M. M. Baldwin, S. F. Etris, J. R. Kanagy, B. L. Lewis, R. Stubbings, and R. L. Young, Jr.

Mr. Lewis reported that his committee did not have an opportunity to formulate the necessary changes in the by-laws.

Because of the difficulty experienced in getting accommodations in New York without a guarantee of a minimum number of room reservations and/or luncheons, it was decided to hold the next meeting at the Benjamin Franklin Hotel in Philadelphia on March 14–15, 1961.

There was considerable discussion about the apparent decline in membership attendance at these meetings. Several possibilities will be explored to increase attendance and to recruit new members. We could use more members to represent tanners and consumers (shoe manufacturers, military procurement, etc.). We will try to get more publicity for the activity of the group and investigate the possibility of having the meeting coincide with some other group or associations with related interests.

In conjunction with the meeting of the International Union of Leather Chemists Societies in Washington, D.C. on August 20–25, 1961, we were approached by Dr. R. G. Mitton, Chairman of the International Union on Physical Testing for a joint meeting preceding the Congress. The meeting will be held on Saturday, August 19, 1961, and all committee chairmen are invited to attend. This will be an excellent opportunity to discuss the European and American aspects of physical testing methods. In order to orient our membership we will ask Mieth Maeser to review the activity of IUPTC at the last Congress that he attended at Munich, Germany, in 1959.

J. NAGHSKI

SUBCOMMITTEE AND SECTION REPORTS

Subcommittee I. Nomenclature, Definitions and Applicability.—Subcommittee I met on Friday, December 9. Doctors Lollar and Filachione were present, while the other three members were absent. No methods were presented to the subcommittee; no action was taken.

It was pointed out that data presented during the meeting of the Joint Committee on Leather to the Deterioration Section disagreed on the reproducibility statement contained in the Spray Rating Test Method which had been previously approved by the Joint Committee. While more data are being secured by the Deterioration Section this disagreement was called to

the attention of the Water Resistance Section for consideration and clarification prior to the recommendation that the tentative method be made official.

It was also pointed out that in those methods where the report of results takes the form of ordinal or scalar values, the use of standard deviation as a measure of precision is inappropriate. However, the sections were urged to secure some numerical data on the reproducibility of these ordinal or scalar values. Emphasis should be placed on the laboratory-to-laboratory variability in the results, skin-to-skin variability in the results, and, where possible, the within-skin variability. While this will not require elaborate statistical analysis, some statement along these lines will aid a person unfamiliar with the method to interpret his results.

The sections were asked to consider terms which might need definition in the dictionary. It was pointed out that the original dictionary had been amended by the inclusion of three definitions pertaining to water resistance. A dictionary reprinting may, therefore, soon be in order, and it might be well to try to make such additional changes as soon as possible.

It was pointed out that the method on the flex testing of upholstery leather developed by Section V had been approved by Subcommittee I as the March 17–18, 1960, minutes record. Hence, this method is in the hands of the Editorial Committee and is not on the agenda of Subcommittee I.

R. M. Lollar
Chairman

Subcommittee II. Sampling and Conditions for Physical Testing.—No report.

Subcommittee III. Physical Testing.

Section 2. Physical Dimensions.—The Physical Dimensions Committee did not meet as a subcommittee but reported instead to the General Assembly of all subcommittees. In connection with requests, the Physical Dimensions Committee is being reconstituted to study (1) factors relating to the development for measuring thickness of soft glovey-type leathers, (2) means for resolving apparent discrepancies in thickness values as measured on the Woburn and Randall Stickney thickness gauges, and (3) the practicability of "snapping" rather than releasing gently the presser foot of the Woburn gauge to conform with trade practice.

Two governmental and one commercial testing laboratories have offered to work on the committee. Representatives of the leather manufacturing

industry which were instrumental in the initiation of the program are being solicited, but so far without success.

Data obtained thus far indicate real differences exist in thickness values of leather depending on whether a Woburn or a Randall Stickney gauge is used and whether the gauge foot of the former is dropped or lowered gently on the leather.

The General Assembly recommended further interlaboratory testing, and on the basis of the data the subcommittee will propose possible methods modification at the next joint meeting.

E. T. STEINER
Chairman

Section 3. Tensile Properties.

1. Methods were discussed, and minor changes will be made in the methods that will clarify their meaning.

2. Extensive testing was done at two different speeds of the testing machines to find out whether our methods could adopt the speed of machines used in methods by the International Union. Data for the test speeds of 4" per minute and 10" per minute show no significant difference. It was approved that both speeds be included in the methods.

3. The size and type wire used in the stitch tear, two-hole method will be studied. Tests will be made on three different diameter wires and on hard and soft types. A government specification FF-C-436a, Clips, Paper refers to clips that are normally used and are available in all laboratories, and they are Type I- Gem-pattern, Size 1. - Large, Class A. - Heavy Duty, Class B, - Light Duty. The specification diameter range is .036 (± .001) and .04 (± .001) and the method requires .038 (± .001). If there is no significant difference, the diameter of the wire will be set as .038 (± .003). This Specification FF-C-436a on the wires will be cited in the method.

4. The testing program to secure data on the precision of the method is nearing completion. When the data are complete and statistical evaluation is accomplished, a statement of precision and accuracy will be written into the method. There is good correlation between two laboratories that are doing the testing, and the differences are not significant. We may be able to have a few methods before the general committee by the next meeting.

R. L. YOUNG
Chairman

Section 4. Measurement of Water Resistance.—One tentative method, D1815-60T, Water Absorption (Static) of Leather, has been published by the ASTM.

The Spray Test Method for Garment Leather is ready for submission to the Editorial Committee.

The two methods for Dynamic Water Resistance (Maeser and Sylflex) are also ready for the Editorial Committee. The statements regarding significance and precision were cleared at the last meeting, and we have now resolved various comments and suggestions which were submitted by committee members.

Dr. Kanagy presented data on the static water absorption obtained in an interlaboratory test on seven different types of leather by both the Kubelka method and D1815-60T. The committee decided not to consider the Kubelka method further, since no advantage in precision could be gained and the apparatus is fragile and expensive. These data are to be presented to *JALCA* for publication for use as future reference.

Some difficulty has been encountered in the interlaboratory test on the dynamic water resistance of heavy leather. We expect that this may be cleared by the time of our next meeting.

R. G. Ashcraft
Chairman

Section 5. Surface Characteristics.

1. A revision of the Tentative Method for Cold Crack of Upholstery Leather was approved by the section to allow the sample to be taped together with pressure sensitive tape as well as with staples. This revision will be submitted to the Applicability Subcommittee, etc. The section considered a further modification of the method to allow the samples to be kept in the chamber between readings and decided to hold up a decision on this until further data were available.

2. Tentative Method for Flex Testing of Finish of Upholstery Leather is now in the hands of the Editorial Subcommittee awaiting action. The Subcommittee on Applicability requested further data on reproducibility before the method can be considered more than tentative.

3. Lightfastness. A new series of tests is being planned to iron out differences between South Florida exposures and fadeometers resulting from the first series of tests. The section also decided to publish the results of these tests provided each of the participating companies agreed.

4. Crock Test. Section agreed on definition of "crock" as being the transfer of colored material from the leather to the test material. The question of evaluation of the results will be studied in relation to various

standard scales. Mr. McKee will write a proposed general method based on the AATCC Crockmeter with the necessary modifications which will be circulated to the section.

5. Scuff Test. Various methods of test are under discussion and preliminary results have been studied by section. A second series of tests using pendulum scuffers, Taber abraders, turntable and needle, etc., will be conducted using some samples of the scuff-resistant leathers.

ROBERT STUBBINGS
Chairman

Section 8. Deterioration.

I. *Perspiration Project.*—Mr. Roddy discussed several methods of exposing specimens to the perspiration solution. It was recommended that a flexing technique be used. Mr. Roddy will prepare the Perspiration Test Method in ASTM form for review by the section.

II. *Dry Cleaning Project*

A. Mr. Olson discussed the results of an interlaboratory test program (Dow Corning and Minnesota Mining). It was concluded:

1. The temperature of dry cleaning between 70°F. and 90°F. does not appear to influence the results.

2. Preconditioning at 50% R.H. and 90% R.H. may have a slight influence on leather response.

3. The spray rating method of measuring leather response is subject to wide variation.

B. Specimens used in the evaluation will be sent to Jerry Ashcraft for spray rating determination. This may give a clearer picture of the expected variability of the spray rating method of test.

C. A second interlaboratory experiment involving a larger number of samples will be undertaken. Maynard Olson will secure six skins. The dry cleaning will be conducted at room temperature, and the preconditioning will be at room temperature.

III. *Laundering Project.*—A report by Mr. Fein on an interlaboratory test was read by Dr. Filachione. The laboratories involved were Walreco, Dow Corning, and Eastern Utilization Research and Development Division.

The use of the gray scale to determine color loss of the leather gave reasonably good agreement between laboratories.

A method of test will be prepared based upon the interlaboratory test data. This will be submitted to the section for comment.

IV. *Heat Stability Project.*—It was concluded by the committee that the twelve lots of leather purchased in early 1959 were satisfactory substrates for additional accelerated aging experiments. Mr. Mann cited work which indicated that under normal storage conditions most leathers do not deteriorate.

V. *Miscellaneous.*—The committee was requested to comment on a communication from Dr. Clara Deasy of the Tanners' Council Laboratory to the ALCA, regarding the elimination of Spore Culture No. 9 from the ALCA Method LI, Resistance of Leather to Growth of Fungi. The section moved to inform the ALCA that in view of the evidence presented by Dr. Deasy, and the experience of several of the section members, it is recommended that Spore Culture No. 9 be eliminated.

It was further suggested that the committee chairman prepare Method LI for submission as an ASTM method of test.

CHARLES F. DUDLEY
Chairman

Section 10. Miscellaneous Physical Properties.—The section chairman reported evaluations carried out on the Navy's Universal Leather Tester on matched treated and untreated sole leather samples showed that butyl treatments substantially improved abrasive resistance and water resistance of the leather. Moreover, leathers commercially treated by the hot dip butyl process performed better than solvent-treated leathers.

The development of an upper leather visual evaluation system and a new shrinkage temperature device for measuring shrinkages above 100°C. was also reported.

MILTON BAILEY

Subcommittee IV. Research.—Attendance included the following subcommittee members:

R. G. Ashcraft
Milton Bailey
N. C. Benrud
J. R. Kanagy
C. W. Mann
W. T. Roddy
G. H. von Fuchs
L. P. Witnauer
M. M. Baldwin (Chairman)

and visitors:

C. F. Dudley
E. M. Filachione
Joseph Naghski
M. H. Olson
S. T. Tu

The current practice of scheduling discussion of the activities of specific sections in meetings for general committee attendance was discussed. One section meeting of this type scheduled for this session did not materialize because of insufficient attendance. If such meetings are to be held in the future they should be given more emphasis. With respect to content of meetings of this type it was suggested that a particular activity of the section be highlighted rather than attempting to review all of the section's activities. It was also suggested that if a specific activity has resulted in suitable information for preparation of a formal paper, such a paper should be presented at an ALCA or ASTM meeting rather than at a Joint Committee on Leather meeting. Also it was suggested that topics such as a discussion of "synthetic leathers" be considered. It was the consensus that the subject of these general discussion sessions be brought up for consideration at the general committee meeting. It was suggested during the Advisory Committee meeting that Mr. Maeser be asked to talk at our next meeting on the international meeting in Munich.

Mr. Roddy reported on his work with the Cenco-Fitch method for measuring the heat conductivity of leather. The results obtained with this method indicate that it warrants consideration by a pertinent section for development as a standard test method. It was suggested that the Miscellaneous Properties section take this under consideration, and Mr. Roddy will send a summary of his work to Mr. Bailey.

Work on an abrasion test for sole leather was reported by Mr. Bailey and Dr. Kanagy. Mr. Bailey's work, in which he used a modified tap tester, is to be reported on more extensively at our next meeting, since there was appreciable interest in the development of a method for measuring sole leather abrasion properties.

The organization meeting of ASTM Committee E-18, Sensory Evaluation of Materials and Products, was reported on by Dr. Naghski; Dr. Tu, who also attended the meeting, made some additional comments. Since the work of E-18 may be pertinent to some of our interests, we plan to keep in touch with their activities. Mr. Roddy indicated that he and Dr. O'Flaherty are planning to attend the next meeting of E-18, which is to be held in Cincinnati on January, 1961.

There was some general discussion of the meaning of tests on leather to the consumer industries and of their need for new methods. It was suggested that a questionnaire be devised to try to determine consumer industry interests in leather testing. Mr. Mann agreed to prepare, with the help of various committee members, a draft of a questionnaire for consideration at our next meeting.

M. M. BALDWIN
Chairman

Subcommittee V.—Editorial.—No meeting.

ABSTRACTS

The Importance of Substances with Active Charges in Retanning Chrome Upper Leather. G. Otto. *Das Leder*, **11**, 217–22 (1960).—Chrome retan leather should have a mild, full feel; elasticity; a fine, tight, elastic grain; and good absorption for finishes. Retanning is affected by many factors, among which are uniformity of distribution of the chrome in the leather, degree of masking of the chrome, degree of neutralization of the leather, nature of the retanning materials, temperature of retanning, and nature of the fatliquoring. For best results the retanning material should penetrate 20–25% of the way through the skin from the grain. If the chrome tannin is too basic or if a strongly filling retanning agent is used, the leather will be spongy, have loose grain, and take up too much finishing material. The following method for rate of water absorption was found to give a measure of leather quality or structural tightness. A capillary tube was drawn fine enough to give a rise of water in it of 4 or 5 cm., the bottom end was cut off clean, and marks were made on the tube 20 mm. and 30 mm. from this end. At a test spot on the leather a small drop of water (2–3 mm. in diam.) was placed, then sucked off with filter paper as soon as the leather darkened, showing it was wet. The capillary, filled with water to about 1 mm. above the upper

mark, was placed on the wet spot, and the time required for the water to fall from the upper to the lower mark was noted. Results were reproducible on the same leather within 3–4%. For different leathers absorption times varied from 5 sec. for vegetable-tanned, anionic-fatliquored, buffed leather to 1750 sec. for chrome-tanned, anionic-fatliquored, unbuffed leather. Tests on a large number of retanned leathers showed that all that had the desirable properties had absorption times lying between 250 and 550 sec. Buffing did not decrease absorption time by more than 10% if the leather was of good quality, and in some instances it even increased it. Leathers with low absorption times had coarse or loose grain and were spongy or at least soft. If absorption time was decreased markedly by buffing, the leather was found to have a coarse break, lacked elasticity, and resembled straight vegetable-tanned leather. By testing before and after buffing it was possible to show the advantages of using materials that changed the electrical charge on the surface of the leather fibers. Such charges are imparted by tanning, dyeing, wetting, and fatliquoring materials. A charged surface will not easily take up more material that has a charge of the same sign but will readily take up material having a charge of opposite sign. A surface charge makes the leather more water-absorbent and tends to open its fiber structure. Alternate treatment with surface-active materials of opposite sign of charge tends to tighten the leather structure. Chrome leather for retanning normally has a weak positive charge, especially if no masking agent was used; masking or neutralizing agents decrease the charge. A positive charge is imparted to the fibers by mineral tannins such as unmasked, basic chromium or aluminum salts, and by cationic soaps and some dyeing agents. Negative charges are imparted by vegetable tannins, lignosulfo acids, syntans, and sulfited, sulfated, or sulfonated oils. Various ordinary anionic chrome retan leathers had absorption times of 200 sec. before and 90–140 sec. after buffing; they did not have tight grain or desirable feel. A rather simple retannage using cationic materials was as follows: Chrome retan leather, tanned with masked chrome salts, was not neutralized but was treated with a low percent of a strongly acid auxiliary syntan which lightly dechromed the leather and made it anionic. It was next treated with a cationic material such as a mixture of highly basic aluminum chloride and a cationic wetting agent or a dye auxiliary, then tanned with an aromatic tannin (vegetable tannin or exchange syntan). It was oiled first with a mixture of water-insoluble and sulfo-group bearing oils, fixed with formic acid, and finally treated with a small amount of a cationic fatliquor. This leather had an absorption time of 260–300 sec. after correction of the grain and of 150–300 sec. before buffing. A firm, full leather could be made by first using an insoluble, cationic resin tannin in a dispersion and retanning with vegetable tannin or syntan. The resin could not easily penetrate the tighter portions of the skin, but was deposited in the looser parts. The anionic retan material was attracted to the cationic resin tannin and so further filled the more open parts of the skin. Production of firm leather by use of some proprietary products is described. Instead of applying certain cationic and anionic materials separately it was found that they could be mixed if they were then used within an hour or so. I.D.C.

The Contribution of Machinery Manufacturers to the Efforts for Economy in the Leather Industry. L. Mägerlein. *Das Leder*, **11**, 213–16

(1960).—Discussion of newer machines and ways for speeding tanning and reducing costs. For example, area-measuring machines now record data and may in the future also stamp the measurement on the skin. To improve performance, studies have been made to determine the proportion of time machines or presses are open for loading and are in actual operation. Future progress will depend on cooperation between tanner and machine manufacturer. To promote safety, manufacturers exchange safety information and do not patent devices. I.D.C.

Particle Size and Tanning Velocity of Vegetable Tannins. G. Reich and H. Legutke. *Das Leder*, **11**, 246–50 (1960).—Particle size of vegetable tannins is important in studies on ways of shortening time of tanning. Determination of molecular or micellar weight by usual methods is not satisfactory; changes in freezing or boiling points, for example, measure principally the unaggregated tannin molecules. Diffusion and osmotic pressure methods are best. Osmotic pressure was determined with a Sorensen-type apparatus (*Z. physiol Chem.*, **106**, 1 [1919]). The rate of movement per unit time of the meniscus in a capillary tube was measured under various back pressures, and a curve showing movement versus pressure was plotted to find the point of no movement, or the osmotic pressure. To calculate micellar weight from osmotic pressure it was necessary to know the degree of solvation of the lyophilic tannin particles, and this was calculated from viscosity measurements and data. Degree of solvation of spruce bark tannin was constant at 5.6 cc. per gram of extract at concentrations between 0.75% and 4% of pure tannin; absorbed water was 4.93 g. per gram of tannin extract. Osmotic pressure in 2% solution at 23°C. was 0.0288 atmospheres, corrected particle weight was 18,760, and particle radius, assuming close spherical packing, was 19.15 angstroms. To avoid errors from soluble substances other than tannin when measuring osmotic pressure, nontannin solution, detannized with hide powder, was put on the outside of the membrane instead of water. Some micellar weights and diameters of particles in 2% extract solutions were as follows: spruce bark, 18,800 and 38.3 resp.; wattle bark, 11,700 and 32.8; oak wood, 6400 and 26.6; quebracho (sulfited), 5200 and 24.7; and chestnut wood, 3500 and 21.6. The particle diameters of vegetable tannins thus vary from 20 to 40 angstroms, and therefore these tannins cannot penetrate to the protofibrils which are separated by distances of from 12 to 17 angstroms. Micellar weight is influenced by several factors. It increased very little as concentration of the tannin solution increased from 1% to 4% for oak, quebracho, and chestnut, but it increased greatly for spruce and wattle, from 5,000 to 20,000 or 30,000. It also increased greatly as pH of the solution decreased or as salt concentration increased. There was a temperature effect, but it could not be measured.
I.D.C.

Interaction between Perfluoro-octanoic Acid and the Fibrous Proteins Keratin and Collagen. M. Feughelman and A. Haly. *Biochimica et Biophys. Acta*, **43**, 550 (1960).—Collagen fibers from rat tail when placed in a saturated aqeous perfluoro-octanoic acid solution at room temperature contract rapidly and swell. X-ray diffraction photographs of collagen fibers treated for 20 min. show a major weakening in the high-angle X-ray diffraction

pattern. Extended treatment (more than 2 days) gave a rubbery material. The PFO apparently enters into the crystalline structure of collagen fiber (keratin also) by its ability to enter the hydrogen-bonded network which stabilizes the crystalline phase. J.M.C.

Condensed Tannins. 6. Biochemical and Stereochemical Interrelationships of (+) Fustin from Black-Wattle Heartwood *(Acacia Mollissima)*. D. Roux and E. Paulus. *Biochem J.*, **77,** 315–20 (1960).—Fisetin and (+) Fustin (2,3 dihydrofisetin) were isolated from the heartwood of *Acacia mollissima* by countercurrent distribution and paper chromatographic techniques. Melting points and optical rotations of enantiomorphous fustins and their racemates are given. The radical distributions of (+) mollisac acidin, (+) fustin and fisetin in wattle heartwood suggest that (+) mollisacacidin is the primary precursor from which (+) fusetin and fisetin are formed by dehydrogenation. J.M.C.

Some Properties of Neutral Salt–Soluble Collagen. 1. J. Fessler. *Biochem J.* **76,** 452–63 (1960).—Isolation of neutral salt–soluble collagen (NSS collagen) was by repeated salt precipitation of extracts of rat skins and rabbit skins. The NSS collagen was separated into three parts: (*a*) fraction A, which reversibly formed fibers at 37°C. in phosphate media at neutral pH, the fibers dissolving on cooling, (*b*) fraction B which did not form fibers at 37°C., (*c*) fraction C which gave fibers at 37°C. which did not dissolve on subsequent cooling. The original material and the separated fractions were investigated by sedimentation, viscosity, and streaming birefringence analyses. No significant differences were found between fractions. Particle weight was estimated at 500,000. No differences in hydroxyproline, proline, or glycine contents of the separate fractions were detected. **2,** *ibid.*, 463–71.—Further examination of fraction B showed that striated fibers typical of collagen could be precipitated by concentrating the polymer phase as well as inorganic buffer phase. Fibers were not formed on warming enriched fraction B in phosphate buffer pH 7.4 (in contrast to ready precipitation of A). At pH 3.7 the specific rotation of B varied with temperature (0°–37°), whereas those of A and C did not. The glycine of fractions A, B, and C had identical specific radioactivities in samples isolated from guinea pigs 6 hr. after injection of C^{14} glycine. The similarity between the effects of temperature upon the solubility behavior of fraction A and the optical rotation of gelatin is pointed out. J.M.C.

Tanning Properties of Dialdehyde Starch. H. Toyoda and T. Komuro. *Bull. Japanese Assoc. Leather Technol.*, **6,** 61–69 (1960) (English summary).—Pieces of goatskin and steerhide were tanned with solutions of dialdehyde starch prepared by electrolytic oxidation of starch under various conditions. Effects of duration of treatment, temperature, pH, concentration, salts, and aging of the solutions on rate and degree of tannage (judged from shrinkage temperature) were studied and compared with HCHO tannage under similar conditions. Tanning power of dialdehyde starch increases with pH value; its tanning power is lower than that of HCHO in acid solution, and in alkaline

solution it is unstable and gradually loses its tanning power on standing at room temperature. Penetration of dialdehyde starch is slower than that of HCHO, and casehardening is likely to occur at higher temperatures or pH values. H.B.M.

Studies on Tanning with Suminagashi. II. Effects of Metal Salts on Sumi Film. H. Kakiyama, H. Kamei, and N. Miyazaki. *Bull. Japanese Assoc. Leather Technol.*, **6,** 70–75 (1960) (English summary); Cf. abstr. *JALCA*, **56,** 46 (1961).—The film is solidified by salts of Hg(II), La, Ce, Cd, and Ba, in addition to the metals previously reported. A method is described for measuring the thermal stability of gelatin gel reacted with metal salt. A test tube containing 10 ml. of 5% gelatin and 1 ml. of 0.1*N* metal salt is kept at 30°C. for 20 hr., then placed in ice water for 30 min. and transferred to a thermostat at 25°–30°C. Time required for the gel to melt is measured. The order of increasing effectiveness in raising the melting point was: Ce - Sr - Cd - La - Ca - Ba, Co - Ni - Ag - Mn, Mg - Zn, Cu - Hg, Fe, Al - Cr. H.B.M

Utilization of Additives in the Salting of Hides. I. Halász. *Bőr- és Cipőtechnika*, **9,** 129–32 (1959); *Hung. Tech. Abstr.*, **12,** 105 (1960).—The effect of bactericidal additives on the conservation of hides has been investigated over a period of several years. From among the chemicals applied (Merpin, silicofluoride, *o*-oxyquinoline, dinitro-orthocresol), sodium silicofluoride yielded the best results. Preservation with a salt mixture containing 2% silicofluoride resulted in hides, pelts and leathers meeting all requirements. The process is now being employed on an industrial scale.

The Effect of Splitting on the Quality of Leather. L. Vermes and I. Malovecz. *Bőr- és Cipőtechnika*, **9,** 141–43 (1959); *Hung. Tech. Abstr.*, **12,** 105 (1960).—Tests have proved that butts of relatively the most uniform thickness can be produced by splitting during the process of tannage. Among the strength characteristics those of tensile strength, tear and stitch-tear resistances deteriorated from splitting. Behavior in respect to water was the reverse of the strength characteristics. The minimum water permeability and water absorption were obtained after splitting during tannage, resistance to water being the lowest with leather split in a finished state. From the viewpoint of water absorption, removal of 30–35% of the hide substance resulted in splits identical with the quality of unsplit material.

Comparison of Methods for Determining the Quantity of Sediments in Tan Liquor. B. Tóth. *Bőr- és Cipőtechnika*, **9,** 180–83 (1959); *Hung. Tech. Abstr.*, **12,** 106 (1960).—Known methods and those which may be taken into consideration for determining the quantity of sediments were investigated and compared. Sedimentation in a graduated cylinder, centrifuging and the method of determining the dry weight before and after sedimentation were compared. A "standard sludge" was prepared for these experiments which was mixed with water in different proportions and the mixture used to establish the reliability of the methods. Experiments with the model sludge

showed that weight or volume determinations by centrifuging could be carried out the most favorably. From among the three types of centrifuges tried, the most suitable was the swinging head-type apparatus of 2000–8000 rpm equipped with speed governor, revolution counter and interchangeable cups of different capacity. Further experiments covered a comparison of various vegetable tanning materials with the synthetic Hungarian products Syntanol Extra OP and Syntanol Extra LF 30 viewed from their proneness to bring about sedimentation at different concentrations.

Utilization of the Mud Formed during the Manufacture of Syntans. J. Lendvai. *Bőr- és Cipőtechnika*, **9**, 154–55 (1959); *Hung. Tech. Abstr.*, **12**, 106 (1960).—Large quantities of mud are formed in the course of producing certain syntans. The mud consists mainly of ammonia alum, gypsum and of insoluble organic substances with considerable amounts of utilizable syntan in the form of enclosures. Methods for utilizing the mud are described, one of which consists in elutriating the mud with water and recovering the alum through crystallization. By another method the mud is used for retanning the chrome chips used in the manufacture of artificial leather.

Improved Bag Tannage with a Blend of Ashwan Bark and Myrobalans. G. N. Hore and A. Ganesan. *Bull Central Leather Research Inst., Madras*, **7**, 112–14 (1960).—Tannage is done with 150% (on fleshed weight) of a 3:1 mixture of ashwan bark and crushed myrobalans. The partially delimed hides are tanned 2 days in pits with leach liquor from ¼ of the tannin mixture, then sewn into bags, filled with the rest of the tannin mixture plus old tan liquor, and tanned 5 days longer. The leather is washed, scoured, treated with a bleaching syntan, oiled, and dried. H.B.M.

A Technique for the Determination of Actual Thickness of Leather-making Substance in Raw Hides. S. K. Mitra and Y. Nayudamma. *Bull. Central Leather Research Inst., Madras*, **7**, 115 (1960).—A cut edge is stained with acid alcoholic resorcin fuchsine which stains the corium violet and the flesh red or magenta. The thickness of the corium is measured. H.B.M.

Leather Dyeing and Staining. G. W. Douglas. *Tanner* (Bombay), **15**, 179–83 (1960).—Descriptive. H.B.M.

Nature of the Linkage of Glutamic Acid in Collagen and Elastin. K. Thomas Joseph and S. M. Bose. *Bull. Central Leather Research Inst., Madras*, **7**, 97–111 (1960).—The great stability of collagen has been ascribed to various cross linkages, one of which may involve the γ-COOH of glutamic acid residues. γ-COOH linkages are resistant to tryptic hydrolysis. Purified collagen (denatured in boiling water to accelerate the enzyme action), alkali-process gelatin, and elastin (from autoclaved *ligamentum nuchae*) were partially hydrolyzed with trypsin or elastase. The hydrolyzates were oxidized with hypobromite, and the resulting succinic acid from glutamic acid not linked

through the γ-COOH was extracted with ether. The extracted solutions were hydrolyzed with HCl to break the γ-COOH bonds, and the hydrolyzed solutions were oxidized and ether-extracted as before. Succinic acid coming from glutamic acid combined through γ-COOH was determined in this second ether extract. The amounts found corresponded to 1.3 mmoles. per 100 g. protein for collagen, but only 0.17 and 0.14 mmoles. respectively for gelatin and elastin. The low value for gelatin indicates that most of the γ-COOH links of collagen are broken by the alkaline gelatin process. Other experiments showed that only 67% of the arginine, and 72% of the lysine, residues of collagen are set free by tryptic hydrolysis, while nearly all these residues are liberated from elastin by successive treatments with elastase and trypsin. Thus it is possible that γ-COOH groups in collagen are bound to part of the arginine or lysine. The small number of γ-COOH links in elastin may be γ-COOH peptides or may involve carbohydrates. H.B.M.

Nature of Tannins in Oak *(Quercus Incane)* and Kaphal *(Myricangi Thunb)*. P. S. Santhanam and S. K. Barat. *Bull. Central Leather Research Inst. Madras*, **7**, 20–22 (1960).—Both these barks contain hydrolyzable tannin only. The oak contained tannin, 17.6%; soluble nontannin, 8.7%; sugar (as glucose), 11.4% (on dry basis); and pH of analytical solution, 4.55. Corresponding values for the kaphal were 36.0%, 12.0%, and 3.3%, and pH 4.5. Titration curves showed that the oak was much better buffered, and therefore mellower. Optimum leaching temperatures were 50°C. for the oak, and 80°C. for kaphal. Leather tanned in the laboratory with the oak was superior to kaphal leather with respect to color and freedom from crackiness, but the degree of tannage of kaphal leather was 111, against only 76 for the oak leather. H.B.M.

Low-Cost Shrinkage-Temperature Tester. T. Gopinath. *Bull. Central Leather Research Inst., Madras*, **7**, 68–70 (1960).—The arrangement is similar to the Theis meter, with hand stirrer, and with the movement of the pulley magnified directly by a pointer and scale instead of through a gear train. H.B.M.

Estimation of Tannase and Certain Oxidizing Enzymes in Indian Vegetable Tanstuffs. W. Madhavakrishna, S. M. Bose, and Y. Nayudamma. *Bull. Central Leather Research Inst., Madras*, **7**, 1–11 (1960).—The enzymes of avaram, konnam, and babul barks, and of myrobalans fruit, divi divi pods, and dhawa leaves, were studied. Tannase was obtained by macerating in ice water, centrifuging with refrigeration, and precipitating the enzyme by dropwise addition of the supernatant liquor to 5 volumes of acetone at –10°C. Tannase was estimated by measuring the liberation of glucose from tannic acid. A mixture of 20 ml. of 1% tannic acid solution and 4 ml. of 1.5% crude tannase solution was digested 24 hr. at 45°C. Tannin was removed by Pb acetate, excess lead was removed with $K_2C_2O_4$, and the glucose content of the deleaded solution was measured by the anthrone method. Polyphenoloxidase, peroxidases, and ascorbic acid oxidase were obtained and estimated by usual procedures. Catalase was looked for, but results were negative. The other 4 enzymes were found in all the 6 tanstuffs in varying quantities—more in

mature than in "tender" growths. Much more tannase was found in the materials containing hydrolyzable tannins, and much more polyphenoloxidase and ascorbic acid oxidase in the condensed tannin materials (the 3 barks). The functions of the enzymes in the synthesis of the tannins are discussed. H.B.M.

Manufacture of Charas Leather by Improved Bag Tanning. S. P. Ghosh and A. Ganesan. *Bull. Central Leather Research Inst., Madras*, **7**, 12–15 (1960).—Air-dried hides are given an alkaline soak, unhaired by painting, relimed 4 days, delimed, and tanned with 100% tanning material (on fleshed weight) consisting of 3 parts babul bark and 1 part myrobalans. The hides are tanned (colored) 3 days in pits with liquor obtained by leaching ¼ of the tanning materials, then sewn into bags which are filled with the remaining tanning material and leach liquor. After 3 days the leather is washed, scoured, oiled on flesh and grain, dried, and staked. **Manufacture of Sole Leather by Improved Bag Tanning Process.** S. P. Ghosh, W. N. Pandy, and A. Ganesan. *Ibid.*, 16–19.—Wet-salted buffalo hides are soaked 6 hr., washed, cut into sides, limed, partially delimed, and tanned as described above using 125% of a 3:1 mixture of babul bark and myrobalans. **Improved Bag Tannage with a Blend of Ashwan and Myrobalans.** G. N. Hore and A. Ganesan. *Ibid.*, 112–14.—See abstract p. 193. H.B.M.

Hydroxyproline and the Shrinkage Temperature of Collagen. J. Bowes and J. Moss. *Nature*, **188**, 1120 (1960).—The authors take exception to the suggestion of B. Rigby and J. Spikes (*Nature*, **187**, 150 [1960]) that rheumatic effects are the result of a structural breakdown of collagen due to temperature effects only. **Hydroxyproline and the Shrinkage Temperature of Collagen.** B. Rigby and J. Spikes. *Ibid.*—The authors reiterate their statement that, since the denaturation and shrinkage of collagen is a time-dependent process, collagen will denature at temperatures below the instantaneous shrinkage temperature, given a sufficiently long time, and that this time will be reduced (at any fixed temperature) if the percentage of hydroxyproline is also reduced. C.L.D.

Chemistry of Protein Fibers. J. Speakman. *J. Textile Inst.*, **51**, 392–407 (1960); abstr. in *J. Soc. Dyers Colourists*, **76**, 702 (1960).—A review (chiefly on wool keratin) with 55 references. C.L.D.

Some Aspects of Fiber Physics. R. Meredith. *J. Textile Inst.*, **51**, 527–37 (1960); abstr. in *J. Soc. Dyers Colourists*, **76**, 702 (1960).—A review on fiber dimensions and density and their optical, electrical, thermal and mechanical properties in relation to stresses and strains, moisture, and heat. C.L.D.

Fundamentals of Fiber Research—A Physicist's Story. W. Astbury. *J. Textile Inst.*, **51**, 515–26 (1960); abstr. in *J. Soc. Dyers Colourists*, **76**, 702 (1960).—A review. C.L.D.

States of Aggregation of High-molecular Compounds. VII. Effect of Temperature on Keratin. R. Fel'dman. *Kolloid. zhur.*, **22,** 351–56 (1960); abstr. in *J. Soc. Dyers Colourists*, **76,** 710 (1960).—The effects of heating and cooling under different conditions of humidity on keratin have been investigated, and coefficients of linear expansion, transition points, degree of contraction, and hysteresis effects have been measured. The state of aggregation of keratin is largely determined by the sorbed moisture content as well as by its prehistory, i.e., previous states and kinetics of their changes. C.L.D.

Lightfastness and Surface Activity of Dyes: The Effect of Alkylation and Sulfonation. C. Giles, G. Baxter, W. Black, N. Macaulay, and S. Rahman. *Textile Research J.*, **30,** 934–43 (1960).—A marked increase in surface activity of an ionic dye tends to decrease lightfastness in normal substrates. A smaller increase in surface activity, e.g., by introduction of a short alkyl chain, usually increases lightfastness. The orientation and number of sulfonate groups affect lightfastness. A more even distribution of such groups or increase in their number in the dye molecule improves the fastness. The effect of normal aggregation of the dyes in the substrate upon their lightfastness is apparently in most cases sufficient to mask differences in their chemical reactivity. The lower the solubility of a dye in a film before setting, the higher is its lightfastness; the higher the solubility, the lower the lightfastness. C.L.D.

Propagation of Excitation Energy in Keratin. A. Haly. *Trans. Faraday Soc.*, **56,** 1252–59 (1960); abstr. in *J. Soc. Dyers Colourists*, **76,** 645 (1960).—Effects of ultraviolet radiation on a wool fiber are found in the near-axis of the fiber. It would be expected that this region would be protected by its sheath of protein material. There appears, therefore, to be a transfer of excitation energy over relatively large distances. It is concluded that this is not due to electron or proton migration but may result from diffusion of photochemical products or more direct transfer of excitation energy by means of exciton diffusion or sensitized fluorescence. C.L.D.

Theory and Practice of Leather Finishing by Pigment Colors in the U.S.S.R. V. Eliseeva. *Kožarství*, **9,** 102–8 (1959); abstr. in *J. Soc. Dyers Colourists*, **76,** 654 (1960).—Resin dispersions penetrate better in leather the smaller their particles, the greater their resistance to salting-out, and the lower the surface tension. The most important factor is the potential difference between the dispersed particles and the leather fibers. A dispersion of negatively charged particles should have pH > isoelectric point of leather if maximum penetration is to be obtained. C.L.D.

Research in the U.S.S.R. Tanning Industry. B. D. Breyev. *J. Soc. Leather Trades Chemists*, **44,** 562–67 (1960).—A survey. S.D.

A Concise Account of Our Present Knowledge of the Structure and Chemical Constituents of Collagen. N. T. Crosby. *J. Soc. Leather Trades Chemists*, **44,** 551–61 (1960).—A review, 42 references. S.D.

Refractive Index Measurement as a Simple Method of Works Control in the Tannery. J. H. Sharphouse. *J. Soc. Leather Trades Chemists,* **44,** 542–51 (1960).—Determination of salt in hides, sulfide in unhairing paints, vegetable tannins in solutions, fatliquor oils, water solubles, and finishes by rapid refractive index measurements are discussed. S.D.

The Hydrotropic Action of Surface-active Materials on Hide Substance. H. Loewe and G. Otto. *Das Leder,* **11,** 251–56 (1960).—Wetting agents, which now are used in many tanning operations, have disadvantages as well as advantages; many damage the hide. In neutral media they swell or dissolve collagen by hydrotropic action, that is, by their attraction for water. Ionic materials have great hydrotropic action, anionic materials more than cationic. Alkylsulfates lower shrinkage temperature (T_s) of hide if the number of C atoms in the molecule is between 8 and 14, and their action is greatest if the hydrophobic part of the molecule is of average length, but it varies with type of chain. Type of collagen is also important, for example, fish skin, which has less hydroxyproline and therefore fewer hydrogen bonds than animal skin, is quite readily attacked by hydrotropic agents. Anionic materials have their greatest action on raw hide and chrome-tanned leathers, whereas cationic materials tend to attack vegetable-tanned leather. Nonionic materials have least effect on collagen. The protective action of surface-active substances against the hydrotropic attack on collagen (calfskin) of wetting agents is shown in the following table:

Materials each 0.05 moles/liter	Swollen Weight	T_s °C.	Dissolved Hide Substance
Blank test with water	100	66	0.2
DBS	148	48	4.4
ANTG	107	64	0.5
HPG	100	67	0.3
DBS + ANTG	114	63	0.4
DBS + HPG	100	66	0.1

The swelling action of p-dodecylbenzenesulfonate (DBS) is entirely repressed by heptylphenylglycolicether (HPG) and to a large extent by an anionic fraction of gambier nontannin (ANTG), which apparently consists of magnesium salts of resorcylic and adipic acids. As the concentration of HPG was increased there was little protective action until a concentration of 0.025 molar was reached, then protective action rapidly approached 100%. The sulfo group of DBS is a strong, localized center of electrical charge and attracts a multiple layer of water dipoles. HPG on the other hand has oxonium dipoles which attract only a thin layer of water dipoles. HPG unites to collagen and, provided its concentration is great enough, prevents attachment of the sulfo groups to the collagen. The hydrotropic action of surface-active agents depends on both a tendency to attach to collagen (determined by the hydrophobic part of the molecule) and a high enough hydration energy (determined by the hydrophilic group). They attach to the long chains of collagen, break hydrogen bonds, and so lower T_s. The hydration centers spread the fibers and swelling results.

The elastic salt-type bonds in collagen can be stretched from about 10 A to 16 A, but if stretched further the bonds break, and strongly hydrated collagen fragments are formed. Cationic wetting agents are weaker than anionic agents as a rule because the sulfate ion is a much more active hydration center than the ammonium ion of quaternary nitrogen compounds. Many anionic wetting agents no longer swell collagen if the medium is changed from neutral to acid, because the cationic groups of collagen are charged and neutralize the strong electromagnetic field of the sulfate ions. The neutralization is permanent because the hydrophobic part of the molecule is fixed to the collagen. The affinity for collagen of the hydrophobic section of the agent modifies its action. At pH 3 phenol swells collagen; p-nitrophenol, 2,4-dinitrophenol, and picric acid do not. At pH 6.5 only 2,4-dinitrophenol has no effect on collagen. For practical use there are alkylsulfonates that have a strong emulsifying action on hide fats but which, if used alone in lime or bate liquors, have a strong hydrotropic action. If used, however, in combination with arylpolyglycols they do not damage the hide. In the mixture the polyglycolic ether coats the collagen so the alkylsulfonate cannot attach to it but directs its action entirely to the fat. I.D.C.

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We visited a side leather tanner the other day who was wrestling with an old problem. He was trying to make a very soft straight chrome tanned side—pasted—in black and colors. As he went up percentage-wise in fatliquor using conventional sulfated oils, he finally got the softness he wanted, but by that time he had gone rag loose in the flank and belly areas of the side. The old dilemma!!

The Polarsol Approach

At this point, one of the Polarsols was introduced into the process as the fatliquor. These esterified oils seem to have the happy property of being able to produce very soft leather but, at the same time, very tight leather—a result not always obtainable with sulfated oils. Using less actual oil, this tanner now produces a butter-soft pasted side that is tight from backbone to belly.

Prove It to Yourself

Smooth, boarded and crushed softie sides for shoe upper and bag are a natural for the Polarsols. How tight can you get? Why not try the Polarsols on your pasted or toggled softie leathers and see for yourself. We'd like to send you a trial sample and complete information on how to use the Polarsols on your softie leathers. Just write or phone.

SEABOARD CHEMICALS, INC.

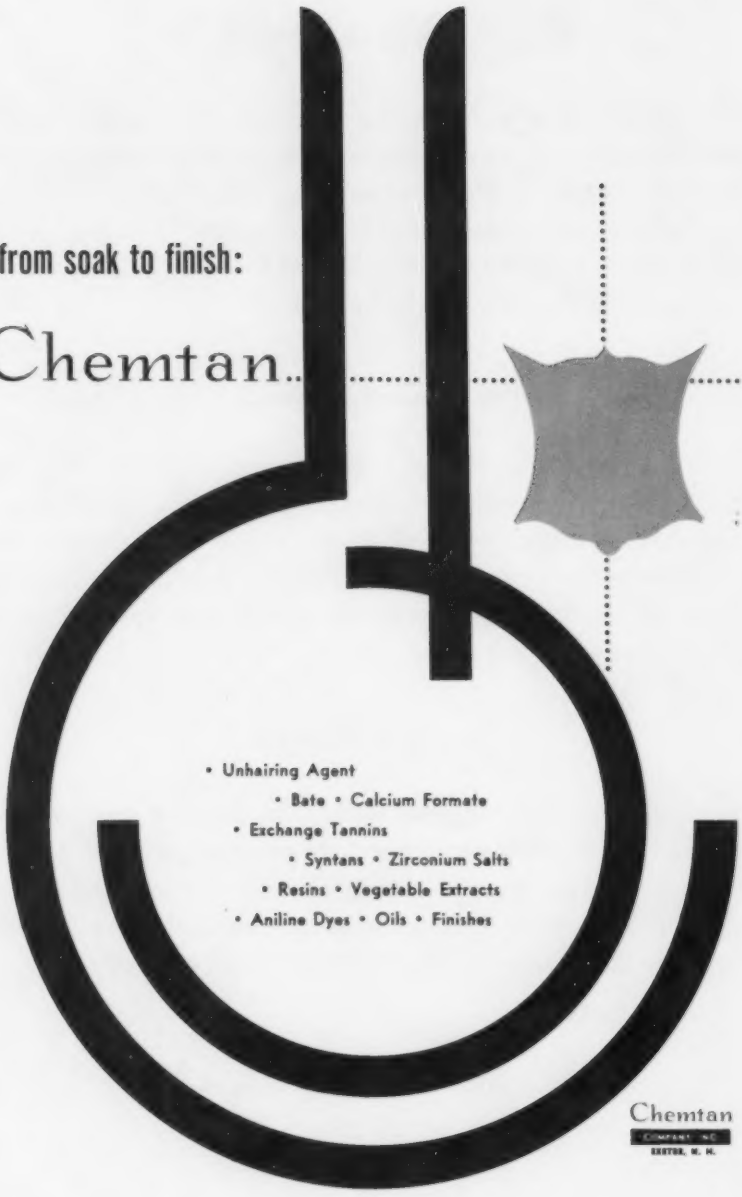
Dept. 41, 30 Foster St.

Salem, Massachusetts

In Canada: Tennant & Michaud, Ltd., Toronto and Montreal.

from soak to finish:

Chemtan..



• Unhairing Agent
• Bate • Calcium Formate
• Exchange Tannins
• Syntans • Zirconium Salts
• Resins • Vegetable Extracts
• Aniline Dyes • Oils • Finishes

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